Estimation of Slow Crack Growth Parameters for Constant Stress-Rate Test Data of Advanced Ceramics and Glass by the Individual Data and Arithmetic Mean Methods

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ESTIMATION OF SLOW CRACK GROWTH PARAMETERS FOR CONSTANT STRESS RATE TEST DATA OF ADVANCED CERAMICS AND GLASS BY THE INDIVIDUAL DATA AND ARITHMETIC MEAN METHODS

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Summary

The two estimation methods, the individual data and the arithmetic mean, were used to determine the slow crack growth (SCG) parameters \( n \) and \( D \) of advanced ceramics and glass from a large number of room- and elevated-temperature constant stress-rate ('dynamic fatigue') test data. For ceramic materials with Weibull moduli \( >10 \), the difference in the SCG parameters between the two estimation methods was negligible; whereas, for glass specimens exhibiting a Weibull modulus of about 3, the difference was amplified, resulting in a maximum difference of 16 and 13 percent, respectively, in \( n \) and \( D \). Of the two SCG parameters, the parameter \( n \) was more sensitive to the estimation method than the other. The coefficient of variation in \( n \) was somewhat greater in the individual data method than in the arithmetic mean method.

BACKGROUND

Advanced ceramics are candidate materials for high-temperature structural applications in heat engines and heat recovery systems. One of the major limitations of ceramic materials in high-temperature applications is delayed failure, where slow crack growth of inherent flaws can occur until a critical size for instability is attained. Consequently, it is important to evaluate slow crack growth behavior accurately so that accurate lifetime prediction of the components is ensured.

For most ceramics and glass, slow crack growth rate \( (v) \) can be expressed by the empirical, power-law relation (ref. 1)

\[
v = \frac{da}{dt} = A\left[\frac{K_I}{K_{IC}}\right]^n
\]

where \( a \) is the crack size, \( t \) is time, \( A \) and \( n \) are the slow crack growth parameters associated with material and environment, \( K_I \) is the mode I applied stress intensity factor, and \( K_{IC} \) is fracture toughness of the material under mode I loading. There are several ways of determining slow crack growth (SCG) of a ceramic material. Typically, the SCG of ceramics is determined by applying constant stress-rate (also called 'dynamic fatigue'), constant stress (also called 'static fatigue' or 'stress rupture') or cyclic loading ('cyclic fatigue') to smooth specimens or to precracked fracture mechanics specimens in which the crack velocity measurements are made. Of these testing methods, constant stress-rate testing has been widely used for decades to characterize SCG behavior of ceramic materials at both ambient and elevated temperatures. The advantage of constant stress-rate testing over other methods lies in its simplicity: Strengths are measured in a routine manner at three to four stress rates by applying the displacement-controlled mode (that is, using a constant crosshead speed) or the load-controlled mode (that is, using a constant

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loading rate). The SCG parameters $A$ and $n$ required for design are simply calculated from a relationship between strength and stress rate (ref. 2). These merits have prompted an effort to establish an ASTM standard for constant stress-rate testing (ref. 3).

In constant stress-rate testing which employs constant crosshead speeds or constant loading rates, the corresponding strength ($\sigma_f$) is expressed (ref. 4)

$$\sigma_f = D(\bar{\sigma})^{1/(n+1)} \quad (2)$$

where $\bar{\sigma}$ is the applied stress rate and $D$ is a parameter which depends on $n$, inert strength (strength with no slow crack growth), fracture toughness, and crack geometry factor. The parameter $A$ in equation (1) can be obtained from $D$ with the appropriate relation (ref. 2). Currently, several statistical methods are available in estimating the SCG parameters $n$ and $D$. These include Weibull median, median deviation, individual (all) data, arithmetic mean, homologous stress, bivariant, and trivariant methods, and so on (refs. 5 to 7). In principle, most of these techniques utilize the least squares, best-fit regression analysis primarily based on equation (2). The maximum likelihood estimation method using either median or individual data has been used by Gross et al. (ref. 7). Each method possesses its own advantages and disadvantages over other methods. However, the parameters to be estimated should converge, independent of estimation method, if a sufficient number of test specimens (>40 per stress rate) is used. It is also important to note that the estimation method should be simple and convenient to use. This is particularly important when a test method including SCG parameter estimation is to be standardized.

Of the estimation methods mentioned above, the individual data and the arithmetic mean methods are simple, convenient, and widely used. Taking the logarithm of both sides of equation (2) yields:

$$\log \sigma_f = l/(n + 1) \log \bar{\sigma} + \log D \quad (3)$$

The least-squares, linear regression analysis of $\log \sigma_f$ (dependent variable) versus $\log \bar{\sigma}$ (independent variable) gives the slope $\alpha = 1/(n+1))$ and the intercept ($I = \log D$) as follows [found in any statistical references]:

$$\alpha = \frac{\sum (\log \sigma_f(\log \sigma_f)) - (\sum \log \sigma)(\sum \log \sigma_f)}{J \sum (\log \sigma)^2 - (\sum \log \sigma)^2} \quad (4)$$

$$I = \frac{\sum \log \sigma_f \sum (\log \sigma)^2 - \sum [(\log \sigma)(\log \sigma_f)] \sum \log \sigma}{J \sum (\log \sigma)^2 - (\sum \log \sigma)^2} \quad (5)$$

where $J$ is the total number of data points. From the slope $\alpha$, $n$ can be determined. The individual data method (IDM) uses each individual strength value and the corresponding stress rate to determine $n$ and $I$. In this case, $J$ is the total number of data points. By contrast, the arithmetic mean method (AMM) utilizes the arithmetic mean value of the individual strengths obtained at a given (averaged) stress rate. Hence, $J$ corresponds to the number of stress rates applied, typically three to four. Because of this, the arithmetic mean method is simpler than the individual data method in terms of computational procedure. It also gives the mean strength values directly in a plot of $\log \sigma_f$ versus $\log \bar{\sigma}$.

The objective of this study is to estimate the statistical reproducibility of the SCG parameters for several ceramics and a glass by using both the individual strength data and the arithmetic mean strength values, in order to compare the two estimation methods. The previously published, ambient and elevated-temperature constant stress-rate ('dynamic fatigue') test data that were determined from eight ceramic materials and one soda-lime glass were utilized for this purpose.
CONSTANT STRESS-RATE ('DYNAMIC FATIGUE') TEST DATA

All of the test data determined from ceramic materials were obtained in uniaxial flexure via four-point configurations; whereas, the test data from soda-lime glass in room-temperature distilled water were obtained in biaxial flexure via ring-on-ring configurations. A total of eight ceramics and one soda-lime glass were used: Five silicon nitrides of NCX34 (1200 and 1300 °C) (ref. 8), GN10 (1300 °C) (ref. 9), NC132 (1100 °C) (ref. 10), SN251 (1371 °C) (ref. 11), and SNW1000 (1300 °C) (ref. 12); one SiC whisker reinforced (30 vol%) silicon nitride (GN10 Si3N4/SiCw) (1300 °C) (ref. 9); one silicon carbide of NC203 (1300 °C) (ref. 13); one 96 wt% alumina (room temperature and 1000 °C) (refs. 10 and 14); and soda-lime glass plates (ref. 6) and disks (ref. 15) (both in room-temperature distilled water). A ring-on-ring biaxial fixture with 22.5 mm loading- and 36 mm support-ring diameters was used for the glass disk specimens. The nominal dimensions of the glass disk specimens were 51 and 3 mm, respectively, in diameter and thickness.

Although for some materials a wide range of stress rates ranging from 0.033 to 3333 MPa/s were used in the actual testing, only the strength data corresponding to four stress rates, typically ranging from 0.033 MPa/s to 33.3 MPa/s, were chosen here for the purposes of consistency and comparison with the other available data. A summary of the resulting plots of \( \log \sigma_f \) versus \( \log \sigma_s \), based on equation (3), for all the test materials is shown in figure I. The individual data points determined at each stress rate were plotted in the figures.

RESULTS AND DISCUSSION

The SCG parameters \( n \) and \( I \) of seven ceramic materials tested at elevated temperatures, estimated by both the individual data and the arithmetic mean methods, are summarized in table I. Table I is for the test conditions of three to four stress rates with three to five specimens per rate. A summary of the SCG parameters as a function of the number of test specimens for two ceramics and soda-lime glass biaxial plate and disk specimens is also shown in table II. The groups of test specimens in table II were taken in groups of five from the test sequence of the raw data (that is, from the testing order) until the total number of test specimens was reached. The tables (I and II) also include the ratios of the SCG parameters \( n \) and \( I \) estimated by the arithmetic mean method to those by the individual data method, which are designated, respectively, as \( r_n \) and \( r_I \).

Figure 2 shows a summary of the SCG parameter \( n \) estimated by both the individual data and the arithmetic mean methods. As can be seen in the plots, little difference in \( n \) between the two estimation methods is found. A more detailed comparison of \( n \) between the two estimation methods was made using the ratio \( r_n \) of \( n \) estimated by the arithmetic mean method to that estimated by the individual data method, as shown in figure 3. The maximum difference in \( r_n \) between the two methods was 2 percent for SN251 Si3N4 (see also table I). Otherwise, the difference is less than 1.7 percent for other ceramic materials. This indicates that the SCG parameter \( n \) can be determined with a reasonably high accuracy either by the individual data method or by the arithmetic mean method for the typical data set of four stress rates with the 3 to 5 test specimens per stress rate. In other words, for a ceramic material with its Weibull modulus of about 10 (typical of most advanced ceramics) the difference in \( n \) between the two estimation methods is negligible for the set of data given in table I.

Figure 4 shows the SCG parameter \( n \) as a function of number of test specimens for NC203 SiC, 96 wt% alumina, and soda-lime glass biaxial plate and disk specimens. The SCG parameter \( n \) varies with the number of test specimens for all the materials. The \( r_n \) ratio is also depicted in figure 5. For the ceramic materials, the maximum difference in \( n \) between the two estimation methods is 5.8 and 0.6 percent, respectively, for NC203 SiC and 96 wt% alumina (see also table II). By contrast, the difference in \( n \) for the soda-lime glass biaxial specimens is appreciable with a maximum difference of 16 and 9 percent, respectively, for the plates and the disk specimens. The difference was reduced to 3.6 and 6 percent, respectively, for the plate and the disk specimens when the respective number of test specimens was increased to 30 and 25 per stress rate. Also, the difference for NC203 SiC was reduced to 2.2 percent when the number of test specimens was increased to 20 per stress rate. A somewhat larger difference for the glass specimens, compared with the ceramics specimens, is primarily due to the low Weibull modulus (=3) of the material. The glass specimens were prepared such that an asreceived, large plate glass was cut into square or circular specimens, annealed at 520 °C for 24 h and then etched in a 20% H2SO4-20% HF-60% H2O solution for 2 min to remove spurious machining and handling damage. The glass specimens thus prepared exhibited a low Weibull modulus of about 2 to 5 (refs. 6 and 15).
The SCG parameter \( I \) estimated by the two methods for the seven ceramic materials tested at elevated temperatures is shown in figure 6. The resulting ratio \( r_I \) of \( I \) estimated by the arithmetic mean method to that by the individual data method is also shown in figure 7 (see also table I). The maximum difference in \( r_I \) between the two estimation methods was about 0.1 percent for SNW1000 Si\(_3\)N\(_4\). This difference gives an actual difference of 0.7 percent in \( D \). It is thus concluded that either the individual data or the arithmetic mean method can be utilized without virtual errors in estimating the SCG parameter \( I \) (or \( D \)) for the set of data given in this example.

The SCG parameter \( I \) of each material, estimated by the two methods, as a function of number of test specimens is depicted in figure 8. The effect of \( r_I \) the number of test specimens is also shown in figure 9, constructed from the data shown in table II. The effect is negligible for both NC203 SiC and 96 wt% alumina with the corresponding maximum difference of 0.2 and 0.01 percent, respectively. However, the difference is amplified for the soda-lime glass specimens, particularly for the plate specimens. The maximum difference is 2.3 and 0.9 percent, respectively, for the glass plate and the disk specimens. This gives an actual difference in \( D \) of about 12.6 and 4.1 percent, respectively. As in \( r_n \) the difference generally decreases with increasing number of test specimens. The greater difference in \( I \) for the glass specimens, compared with the ceramics specimens, is again due to its low Weibull modulus (=3). It is also noted that the difference between the two estimation methods is always lower in \( I \) than in \( n \).

The fact that the difference between the two estimation methods for the constant stress-rate test data is more dominant in \( n \) than in \( I \) gives again an insight into the necessity of accurate estimation of \( n \). Lifetime \( (t_f) \) of a ceramic component for a given applied load is expressed as follows (ref. 2):

\[
t_f = f(G)\sigma^{-n}
\]

where \( \sigma \) is the applied stress and \( f(G) \) is the parameter associated with \( n \), inert strength, fracture toughness and crack geometry factor. Because of this functional form, lifetime of a ceramic component is strongly dependent on \( n \). Therefore, the accurate determination of \( n \) is of greater importance (than \( I \)) if accurate lifetime prediction of the component is to be ensured.

The statistical reproducibility of the SCG parameter \( n \) between the two estimation methods can be examined by determining the coefficients of variation in \( n \), \( CV(n) = SD(n)/n \) with \( SD(n) \) being standard deviation of \( n \). The resulting plot of \( CV(n) \) for the seven ceramic materials tested at elevated temperatures, estimated based on table I, is shown in figure 10. Except for SN251 Si\(_3\)N\(_4\), \( CV(n) \) was found to be somewhat (about 0.05 on average) greater in the individual data method than in the arithmetic mean method. Figure 11 shows the coefficients of variation in \( n \) as a function of number of test specimens for NC203 SiC, 96 wt% alumina, and soda-lime glass. This figure, like the results of figure 10, shows that overall \( CV(n) \) in general, is greater in the individual data method than in the arithmetic mean method. The difference in \( CV(n) \) between the two estimation methods is most dominant for the small number of test specimens (<10) for NC203 SiC and the soda-lime glass biaxial plates. The difference, however, becomes negligible with increasing number of test specimens (≥20), resulting in improved statistical reproducibility.

Based on the above results, it can be stated that the difference in the SCG parameters between the two estimation methods depends mainly on Weibull modulus of the material, as the statistical reproducibility does (ref. 16). Either the individual data or the arithmetic mean method can be used with a little error (about 2 percent maximum) to estimate the SCG parameters \( n \) and \( I \) (or \( D \)) for a data set similar in this example, provided that the Weibull modulus is greater than about 10. This is applicable to most properly machined, advanced ceramics since, in general, they exhibit a Weibull modulus ≥10. For a material exhibiting a high Weibull modulus ≥20, no difference in either \( n \) or \( I \) is expected, as evidenced by the 96 wt% alumina specimens: the maximum difference was found to be 0.6 percent and less than 0.1 percent, respectively, for \( n \) and \( I \) (see tables I and II). By contrast, for a material such as the soda-lime glass which exhibited a low weibull modulus of 3, special care should be taken in estimating the SCG parameters. The coefficient of variation in \( n \), \( CV(n) \), is somewhat higher in the individual data method than in the arithmetic mean method. The difference in \( CV(n) \) between the two methods becomes insignificant with increasing number of test specimens (≥20). Based on the applicability of a wide range of Weibull modulus, as well as the result of \( CV(n) \), the least-square, best-fit regression analysis using the individual data points, that is, the individual data method, is preferred in view of its unbiased nature.
CONCLUSIONS

The maximum differences in the SCG parameters $n$ and $I$ between the individual data and the arithmetic mean methods were 2 and 0.1 percent, respectively, for ceramic materials of Weibull modulus $\geq 10$ (with a set of 4 stress rates and 4 to 5 specimens per stress rate). The difference was greater for the soda-lime glass specimens whose Weibull modulus is about 3; the difference in $n$ and $I$ were 13 and 4 percent, respectively. The difference, however, decreased with increasing number of test specimens. Also, the difference between the two estimation methods was more dominant in $n$ than in $I$, emphasizing the importance of accurate estimation of the SCG parameter $n$. In general, the coefficient of variation in $n$ was somewhat greater in the individual data method than in the arithmetic mean method, indicating that the arithmetic mean method tends to bias the SCG parameter $n$, as compared with the individual data method. The individual data method is generally recommended in view of this behavior.

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REFERENCES


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<th>Weibull modulus, approximately</th>
<th>Number of stresses</th>
<th>Number of specimens per rate</th>
<th>SCG parameter ( n )</th>
<th>SCG parameter ( I (\log D) )</th>
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<td>1</td>
<td>NCX34 Si3N4:1200 °C</td>
<td>10</td>
<td>4</td>
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<td>4</td>
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<td>19.78(5.84)</td>
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<td>4</td>
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<td>31.64(6.96)</td>
<td>31.91(5.36)</td>
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*The numbers in the parentheses indicate ± one standard deviation.*
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<td>12.4 to 6.3</td>
<td>4</td>
<td>15</td>
<td>0.58 (±0.12)</td>
<td>0.73 (±0.04)</td>
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<tr>
<td></td>
<td>20.5 to 5.0</td>
<td>4</td>
<td>20</td>
<td>0.51 (±0.12)</td>
<td>0.70 (±0.03)</td>
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<tr>
<td>98% w/w Al₂O₃-3H₂O (ref. 6)</td>
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<td>4</td>
<td>10</td>
<td>0.66 (±0.07)</td>
<td>0.81 (±0.03)</td>
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<tr>
<td>98% w/w Al₂O₃-3H₂O (ref. 6)</td>
<td>20</td>
<td>4</td>
<td>10</td>
<td>0.66 (±0.07)</td>
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The numbers in the parentheses indicate ± one standard deviation.
Figure 1.—A summary of constant stress-rate testing results from various ceramics and glass: (a) NCX34 silicon nitride (1200 and 1300 °C) [8]; (b) GN10 SiC whisker-reinforced (30 vol%) silicon nitride (1300 °C) [9]; (c) GN10 silicon nitride (1300 °C) [9]; (d) NC132 silicon nitride (1100 °C) [10]; (e) 96 wt% alumina (1000 °C) [10]; (f) SN251 silicon nitride (1371 °C) [11]; (g) SNW1000 silicon nitride (1300 °C) [12]; (h) NC203 silicon carbide (1300 °C) [13]; (i) 96 wt % alumina (room-temperature water) [14]; (j) soda-lime glass biaxial plates (room-temperature water) [6]; (k) soda-lime glass biaxial disks (room-temperature water) [15].
Figure 1.—Continued. (e) 96 wt% alumina (1000 °C) [10]; (f) SN251 silicon nitride (1371 °C) [11]; (g) SNW1000 silicon nitride (1300 °C) [12]; (h) NC203 silicon carbide (1300 °C) [13].
Figure 1.—Concluded. (i) 96 wt % alumina (room-temperature water) [14]; (j) soda-lime glass biaxial plates (room-temperature water) [6]; (k) soda-lime glass biaxial disks (room-temperature water) [15].
Figure 2.—A summary of the slow crack growth parameter $n$ estimated by both the individual data and the arithmetic mean methods for seven ceramic materials tested at elevated temperatures.

Figure 3.—The ratio ($r_n$) of $n$ estimated by the arithmetic mean method to that by the individual data method for seven ceramic materials tested at elevated temperature.
Figure 4.—The slow crack growth parameter \( n \) as a function of number of test specimens, estimated by both the individual data method and the arithmetic mean method: (a) NC203 SiC (1300 °C) [13]; (b) 96 wt % alumina (room-temperature water) [14]; (c) soda-lime glass biaxial plates (room-temperature water) [6]; (d) soda-lime glass biaxial disks (room-temperature water) [15].
Figure 5.—The ratio \( r_n \) of \( n \) estimated by the arithmetic mean method to that estimated by the individual data method for four test materials, as a function of number of test specimens.

Figure 6.—The slow crack growth parameter \( I \) estimated by both the individual data and the arithmetic mean methods for seven ceramic materials tested at elevated temperatures.

Figure 7.—The ratio \( r_I \) of \( I \) estimated by the arithmetic mean method to that estimated by the individual data method for seven ceramic materials tested at elevated temperatures.
Figure 8.—The slow crack growth parameter I as a function of number of test specimens, estimated by both the individual data and the arithmetic mean methods: (a) NC203 SiC (1300 °C) [13]; (b) 96 wt % alumina (room-temperature water) [14]; (c) soda-lime glass biaxial plates (room-temperature water) [6]; (d) soda-lime glass biaxial disks (room-temperature water) [15].
Figure 9.—The ratio ($r$) of $I$ estimated by the arithmetic mean method to that by the individual data method for four test materials, as a function of number of test specimens.

Figure 10.—A summary of the coefficient of variation in $n$, $CV(n)$, estimated by both the individual data and the arithmetic mean methods for seven ceramic materials tested at elevated temperatures.
Figure 11.—The coefficient of variation in $n$, $CV(n)$, as a function of number of test specimens, estimated by both the individual data and the arithmetic mean methods: (a) NC203 SiC (1300 °C) [13]; (b) 96 wt% alumina (room-temperature water) [14]; (c) soda-lime glass biaxial plates (room-temperature water) [6]; (d) soda-lime glass biaxial disks (room-temperature water) [15].
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**SUPPLEMENTARY NOTES**
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**ABSTRACT**
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**SUBJECT TERMS**
Constant stress-rate ('dynamic fatigue') test; Advanced ceramics; Flexure testing; Slow crack growth parameters; Slow crack growth

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