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Quantitative analysis of delayed fracture observed in stress rate tests on brittle materials

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Delayed fracture in brittle materials may be demonstrated by observing the change in bend strength over a range of constant stress rates to failure. A simple technique has been developed to analyse this behaviour making efficient use of the experimental data. Based on a model incorporating theories of stress corrosion and brittle fracture, with Weibull statistics, the technique provides estimates of relevant parameters using the method of maximum likelihood. Confidence intervals of estimates, the significance of any observed rate effect, and the validity of the model are also assessed. The technique is demonstrated by applying it to data from bend tests on soda-lime glass and WC–Co materials.

1. Introduction

Some recent studies of the effects of stress corrosion in tungsten carbide alloys containing cobalt as a binder phase (WC–Co), have included the measurement of bend strength under different loading rates [1]. Because most commercial grades of WC–Co materials exhibit brittle behaviour the bend strength is influenced by the distribution of flaws through the material in addition to any stress corrosion effect which might be present. It is therefore necessary for both factors to be considered in a quantitiative analysis of stress corrosion.

The simplest method of analysis is to compare the mean strength of a number of specimens at one rate of loading with those obtained at other loading rates (see for example Davidge *et al.* [2], Braiden *et al.* [3]). However, materials like WC–Co are expensive to obtain and prepare, so that the number of specimens available for testing may be relatively small. Consequently possible errors in the mean strength can be considerable. It is obvious that a more efficient method of data analysis is required. More specifically, error due to initial grouping such as calculation of means, must be removed, and the original data employed within a single analysis.

Trustrum and Jayatilaka [4] have used an analysis of this type to estimate a brittle fracture parameter, the Weibull modulus, "m". Following their example a simple technique is reported here which enables estimates to be obtained of both the brittle fracture and the time dependent strength parameters from stress rate data, within a single process. Valid statistical procedures are employed which are based on the method of maximum likelihood.

2. Development of failure model

2.1. Theory

It is now well established that crack growth in a brittle solid subject to stress corrosive conditions may be described by a relationship between K_I, the plane strain stress intensity factor in the crack opening mode and v, the crack growth velocity. This relationship may be written as

$$v = AK_I^n, \quad (1)$$

where A and n are constants, n being often referred to as the stress corrosion parameter.

Further, K_I may be related to the applied stress, σ, through the linear elastic fracture mechanics relationship

$$K_I = Y \sigma a^{1/2}, \quad (2)$$

where Y is a geometric constant and a is some theoretical crack length.

Evans and Johnson [5] have combined these approaches and have hence derived an expression relating the applied stress at fracture σ_f to a constant stress rate $\dot{\sigma}$ employed to raise the stress to the point of fracture, and a theoretical flaw size at the start of loading a_i, namely

$$\sigma_f = \left[\frac{2(n+1)\dot{\sigma}}{AY^n(n-2)a_i^{(n-2)/2}}\right]^{1/(n+1)} \quad (3)$$

Rewriting Equation 3 in logarithmic form

$$\log(\sigma_f) = \frac{1}{(n+1)}\log(\dot{\sigma}) + \left[\frac{n-2}{n+1}\right]\log\left(\frac{1}{a_i^{1/2}}\right) + \log C' \quad (4)$$

where

$$C' = \left[\frac{2(n+1)}{AY^n(n-2)}\right]^{1/n+1} = \text{constant}.$$

Thus the fracture stress is seen to depend upon a nominal constant, the time dependent loading, and the theoretical initial flaw size, the influence of each being controlled by the stress corrosion parameter, n.

2.2. Statistical representation of random variable, a_i

The measurement of n from stress rate data is impeded by the unknown quantity a_i. This is the length of a theoretical initial crack used to represent the complex system of flaws within a real material. Because of their microscopic size, their diversity of type, and complex interaction with others, analysis of real material flaws cannot be used to obtain a value of a_i in the majority of cases. However, the problem may be overcome by considering the material behaviour under a different set of conditions.

If the material is loaded to failure in such a way that stress corrosion is prohibited from acting, say in an inert environment, then the inert strength, σ_I, may be related to the theoretical initial flaw size, a_i, and the fracture toughness (or critical stress intensity factor) of the material, K_{Ic}, such that

$$K_{Ic} = Y\sigma_I a_i^{1/2}, \quad (5)$$

where Y as before is a geometrical constant.

Combining Equations 4 and 5 to eliminate a_i produces

$$\log(\sigma_f) = \frac{1}{(n+1)}\log(\dot{\sigma}) + \frac{(n-2)}{(n+1)}\log(\sigma_I) + \log(C'') \quad (6)$$

where

$$\log(C'') = \log(C') + \frac{(n-2)}{(n+1)} \times \log\left(\frac{Y}{K_{IC}}\right) = \text{constant}.$$

Now the unknown quantity is the inert strength σ_I. Taking material flaws to be randomly distributed, their effect on strength indicates that σ_I may be considered a random variable which represents the brittle strength of the material in the absence of any corrosive influence.

A statistical model proposed by Weibull [6] is commonly employed to characterize the distribution of brittle strength [7].

The form of the distribution of any random variable, X, may be specified by means of the cumulative distribution function (cdf), F, which when evaluated at any number x, gives the probability that a randomly chosen X does not exceed x, i.e.

$$F(x) = P\{X \leqslant x\}.$$

In this case, assuming a Weibull distribution for the random variable σ_I,

$$P\{\sigma_I \leqslant x\} = 1 - \exp\left[-\left(\frac{x}{\sigma_0}\right)^m\right] \quad (7)$$

where σ_0 and m are constant parameters, controlling, respectively, the scale and shape of the distribution.

Equations 6 and 7 together provide a model incorporating both the stress rate influence, and the variability in the fracture stress data that is to be analysed.

2.3. Analysis of the model

Equations 6 and 7 may be combined by first writing

$$Z = m \log\left(\frac{\sigma_I}{\sigma_0}\right). \quad (8)$$

The cdf of Z may be obtained from Equation 7 such that

$$P\{Z \leqslant x\} = P\left\{\sigma_I \leqslant \sigma_0 \times \exp\left(\frac{x}{m}\right)\right\}$$
$$= 1 - \exp(-e^x). \quad (9)$$

This probability distribution, with no parameters, is known as the Gumbel or "extreme-value" distribution [8].

The elimination of σ_I from Equation 6, using Equation 8 gives

$$\log(\sigma_f) = \frac{1}{(n+1)}\log(\dot{\sigma}) + B + \frac{(n-2)}{m(n+1)}Z, \quad (10)$$

where

$$B = \log(C'') + \frac{(n-2)}{(n+1)}\log(\sigma_0) = \text{constant}$$

Equation 10 demonstrates that the model predicts a linear regression of $\log \sigma_f$ on $\log \dot{\sigma}$, the slope of which is $1/(n+1)$. The intercept, B, involves a number of unknown parameters, all constant, and the final term, involving the random variable Z, represents the variability in σ_f.

The distinctive features of this model are:

(1) The "error" distribution is "extreme-value" rather than the more commonly encountered Normal or Gaussian. In consequence, the standard least squares theory is not applicable.

(2) The "errors" in the fracture strength data are more properly systematic random variation caused by the variability in material flaws, the size of which cannot be measured. Thus, these errors are not expected to be negligible, and a close fit of a straight line to the ($\log \dot{\sigma}$, $\log \sigma_f$) data will not be obtained. In this context, measurement error may be neglected.

3. Use of the model to estimate unknown parameters, *n*, *m*, *B*

3.1. Estimation of parameters using the method of maximum likelihood

Although the least squares method of linear regression is not applicable in this case, another standard statistical procedure – the method of maximum likelihood estimation – is valid. It may be used to obtain not only estimates of the unknown parameters in the model (given by Equation 10) but also confidence limits for them (see [8]).

Consider a sample of N observations, (X_i, Y_i) where

$$X_i = \log(\dot{\sigma}), \quad Y_i = \log(\sigma_f) \qquad \text{for the } i\text{th observation,}$$

where $i = 1, 2, 3 \ldots N$.

From Equation 10,

$$Y_i = \frac{1}{(n+1)}X_i + B + \frac{(n-2)}{m(n+1)}Z_i.$$

For temporary notational convenience, this may be written

$$Z_i = rX_i + sY_i + t$$

where
$$r = -m/(n-2)$$
$$s = m(n+1)/(n-2)$$
$$t = -mB(n+1)/(n-2) \quad (11)$$

The cdf of Y_i for a given $X_i = x_i$ (i.e. a fixed preset stress rate) is given, from Equation 9, by

$$P\{Y_i \leqslant y_i\} = P\{Z_i \leqslant (rx_i + sy_i + t)\}$$
$$= 1 - \exp[-\exp(rx_i + sy_i + t)].$$

The probability density function (pdf) of Y_i, $g(y_i)$ is the derivative of the cdf. Thus

$$g(y_i) = \exp[-\exp(rx_i + sy_i + t)] \times \exp(rx_i + sy_i + t) \times s.$$

The likelihood is the product of these terms over the whole sample so that the log-likelihood, or support, S, is given by

$$S = \log \prod_{i=1}^{N} g(Y_i)$$
$$= \sum_{i=1}^{N} \log g(Y_i)$$
$$= \sum_{i=1}^{N} [(rX_i + sY_i + t) - \exp(rX_i + sY_i + t) + \log s]$$
$$= \sum_{i=1}^{N} [(r \log \dot{\sigma}_i + s \log \sigma_{fi} + t) - \exp(r \log \dot{\sigma}_i + s \log \sigma_{fi} + t) + \log s].$$

The maximum likelihood estimates of r, s, t are obtained by maximizing S. Exact formulae are not available, but the maximization can be carried out easily using a standard numerical technique. The estimates of n, m, B may be obtained using Equation 11.

3.2. Confidence in estimated parameter values

The values assigned to n, m, B at the end of the maximization procedure are only estimates. Thus an indication of the confidence that may be placed in them is required. To this end, a likelihood ratio test may be employed (see [8]). Suppose that S_r is the maximum support obtained when the parameters n, m, B are restricted by one fixed constant. If S is the maximum unrestricted support, then

$$\chi^2 = 2(S - S_r)$$

has approximately a χ^2 distribution on one degree of freedom, under the null hypothesis that the constraint actually holds [8].

For example, if the constraint is that $n = n_0$, where n_0 is some preassigned number, and if the maximum support under this restriction is denoted by S_{n_0} then $2(S - S_{n_0})$ has a χ^2 distribution with one degree of freedom if $n = n_0$. The upper 5% point of this distribution is 3.84, so that the collection of values of n_0 such that

$$2(S - S_{n_0}) \leqslant 3.84$$

provides a 95% confidence interval for n.

A similar procedure may be adopted to obtain confidence intervals for m and B. If simultaneous confidence statements about two or three of the parameters are required, then the resulting χ^2 distribution will have two or three degrees of freedom respectively.

3.3. Significance of rate effect

Sometimes it may be necessary to show that the fracture stress is being influenced by the stress rate to a significant degree, so that any apparent correlation between the two is not merely an effect of sampling. In this case, the null hypothesis is that the fracture stress is not influenced by the stress rate, so that a single constraint applies, namely that $n = \infty$. If S, S_∞, are the unrestricted, and restricted supports respectively, then

$$\chi^2 = 2(S - S_\infty) \qquad (12)$$

Whether there is significant departure from the null hypothesis that no rate effect exists is assessed by comparing χ^2 calculated using Equation 12 with the distribution of χ^2 on one degree of freedom given in standard tables.

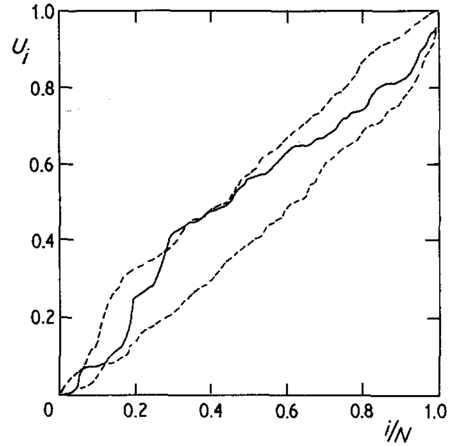

Figure 1 Probability integral transform, U_i, of residual Z_i plotted against i/N ($i = 1, 2, 3 \ldots N$). Dotted lines, envelope of 36 artificial data sets. Solid lines, soda-lime glass.

4. Application to experimental data

Although the above procedure was developed to analyse the fracture strength–stress rate behaviour of WC–Co materials, it was first applied to data from similar tests on abraded soda-lime glass. The maximum likelihood estimates of n and m for both the glass and WC–Co materials are given in Table I.

The maximum likelihood estimates of n for the glass compare favourably with values of n obtained from crack propagation studies using the double torsion configuration, and with values reported in the literature [9] from tests on comparable

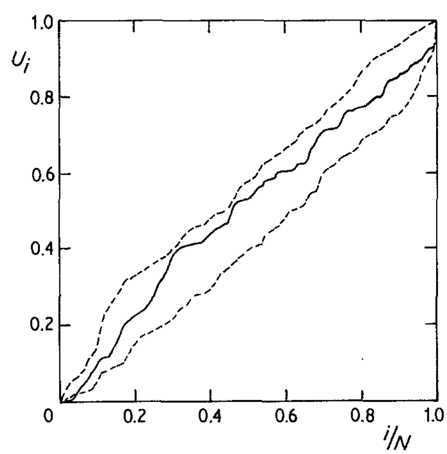

Figure 2 See Fig. 1 for details. Solid line, WC–6 wt% Co (1).

TABLE I Maximum likelihood estimates of m and n, related confidence intervals and the significance of observed rate effect using bend strength data from stress rate tests in laboratory conditions on soda-lime glass and WC–Co materials.

Material (wt% Co)	No. of specimens tested	Range of stress rates ($MN\,m^{-2}\,sec^{-1}$)	No. of different stress rates	ML estimate of n	95% conf. interval for n	ML estimate of m	95% conf. interval for m	Significance, P of rate effect‡
Soda-lime glass (abraded)	68	4–260	7	19	14–28	8.6	7.2–10.2	5×10^{-8}
WC–6 wt% Co (1)*	60	0.1–1000	5	110	46–†	6.5	5.3–7.8	0.12
WC–6 wt% Co (2)*	60	0.1–1000	5	34	24–56	6.8	5.6–8.1	7×10^{-6}
WC–13 wt% Co (1)	58	0.1–1000	5	93	50–620	9.1	7.3–11.2	0.023
WC–13 wt% Co (2)	58	0.1–1000	5	120	59–1100	10.0	8.0–12.2	0.028
WC–16 wt% Co (1)	60	0.1–1000	5	45	34–70	8.8	7.2–10.6	2×10^{-6}
WC–16 wt% Co (2)	60	0.1–1000	5	110	64–330	12.4	9.9–15.0	0.0047

*Surface finish of specimens: (1), ground; (2), diamond polished.

†No finite upper limit exists.

‡P = probability of rate effect being at least as large as that observed when the null hypothesis is true (conventionally, $P < 0.05$ is termed "significant").

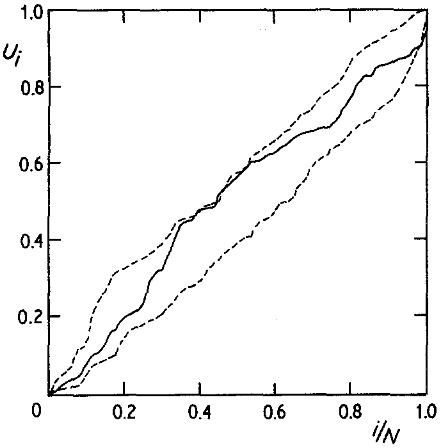

Figure 3 See Fig. 1 for details. Solid line, WC–6 wt% Co (2).

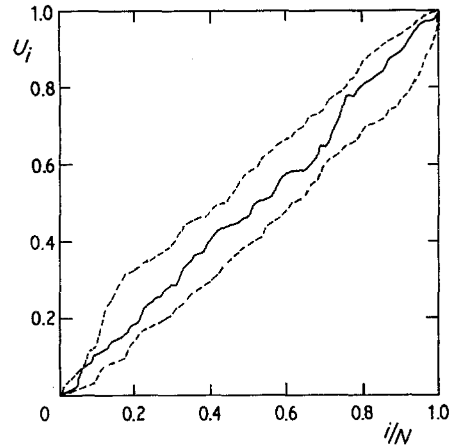

Figure 5 See Fig. 1 for details. Solid line, WC–13 wt% Co (2).

materials. The range of measured n was between 15 and 19.

Double torsion tests were also performed on WC–Co materials containing WC–6 wt% Co and WC–16 wt% Co. Values of n lying between 90 and 140, and 70 and 360, were recorded for these materials respectively. Again these results lie in a similar range of the maximum likelihood estimates.

Neither a description of the experimental techniques employed, nor a discussion of the results will be given here since they are beyond the scope of this report. These details may be found in [1].

5. Assessing the validity of the model

Up to this point, the validity of the model and in particular, the Weibull assumption has not been questioned. Some attempt to do so may be made by examining the residuals, Z_i, in Equation 10, which may be expressed in the form

$$Z_i = \frac{m(n+1)}{(n-2)}\left[Y_i - \frac{X_i}{n+1} - B\right].$$

Z_i is evaluated for each $(X_i, Y_i) = [\log(\dot{\sigma}), \log(\sigma_f)]$, given the estimated values of m, n, B. If the Weibull assumption is correct, then Z_i, $(i = 1, 2, 3 \ldots N)$ should appear to follow approximately the extreme-value distribution [8]. This may be checked graphically by first transforming the Z_i using the probability integral transform

$$U_i = 1 - \exp(-e^{Z_i}).$$

The resulting U_i $(i = 1, 2, 3 \ldots N)$ should be approximately uniformly distributed in the interval $(0, 1)$. If these N values are ordered in magnitude

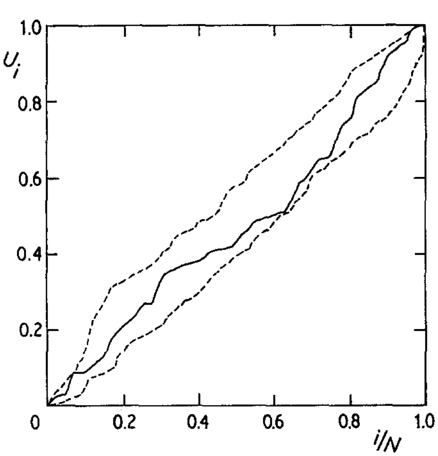

Figure 4 See Fig. 1 for details. Solid line, WC–13 wt% Co (1).

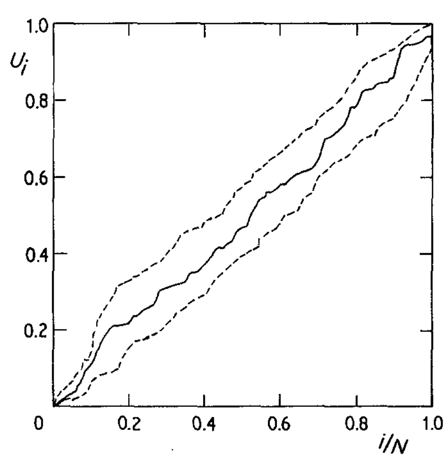

Figure 6 See Fig. 1 for details. Solid line, WC–16 wt% Co (1).

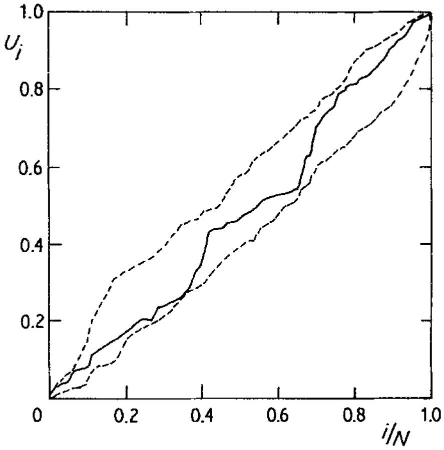

Figure 7 See Fig. 1 for details. Solid line, WC–16 wt% Co (2).

and then plotted against i/N ($i = 1, 2, 3 \ldots N$) the points should lie close to the 45° line from (0, 0) to (1, 1).

Obviously, sampling fluctuations tend to generate departures from this ideal form. If the discrepancies yielded by the data are large, then this would indicate the Weibull model was unsatisfactory. To assess the significance of the discrepancies, a Monte Carlo technique has been used [8].

Thirty-six artificial data sets of the same size ($N = 60$) were constructed with pseudo-random numbers, using the model defined by Equations 6 and 7. The parameters m, n, B were estimated using the maximum likelihood method and the graphs displaying the standardized residuals plotted as for the real data. It may be shown (see [8]) that the distribution of the residuals does not depend

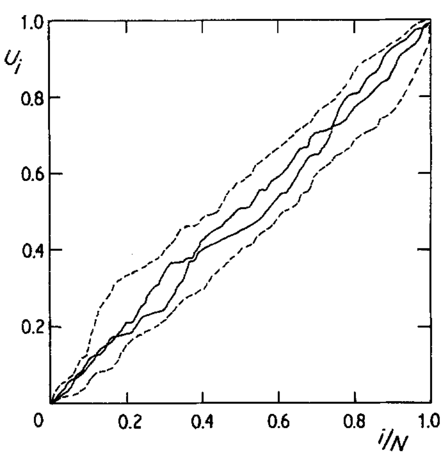

Figure 8 See Fig. 1 for details. Solid lines, two examples of artifical data sets.

on the values of the parameters m, n, B so it is immaterial what values are chosen for the simulation.

The quality of the fit to a 45° line was assessed visually for each artificial data set and compared with the corresponding curves for the real data sets. The results are summarized in Figs 1 to 7 showing the graphs for the seven data sets superimposed on the envelope of the 36 artificial data sets. Examples of graphs for two artifical data sets are given in Fig. 8.

Since each artificial data set was constructed using the Weibull model, any discrepancies from the 45° line originate solely from sampling fluctuations. Curves for the real data sets lie almost entirely within the envelope of the 36 artificial data sets, providing no justification for rejecting the Weibull hypothesis.

The Monte Carlo method may be extended to provide formal tests of significance but these seem unnecessary here. It should be noted that the same method of estimation (maximum likelihood) has been used in assessing the validity of the model as was used in the original estimation of parameters from the real data sets.

6. Conclusions

A simple technique has been developed for the efficient analysis of delayed fracture effects in data obtained from bend tests performed at a range of stress rates.

The fracture model, incorporating theories of stress corrosion and brittle fracture, with Weibull statistics, has an "extreme value" error distribution. Parameter estimation is accomplished using the method of maximum likelihood.

The technique obtains:

(1) the significance of any observed rate effect

(2) estimates of the stress corrosion and brittle fracture parameters

(3) confidence intervals for these parameters

(4) an assessment of the validity of the model.

References

1. B. D. WRIGHT and P. M. BRAIDEN, TIPTOM Conference 1981.
2. R. W. DAVIDGE, J. R. McLAREN and G. TAPPIN, *J. Mater. Sci.* 8 (1973) 1699.
3. P. M. BRAIDEN, R. W. DAVIDGE and R. AIREY, *J. Mech. Phys. Solids* 25 (1977) 257.
4. K. TRUSTRUM and A. JAYATILAKA, A. de S., *J. Mater. Sci.* 14 (1979) 1080.
5. A. G. EVANS and H. JOHNSON, *ibid.*, **10** (1975) 214.

6. W. WEIBULL, *J. Appl. Mech.* **18** (1951) 293.
7. D. G. S. DAVIES, *Proc. Brit. Ceram. Soc.* **22** (1973) 429.
8. D. A. S. FRASER, "Probability and Statistics", (Duxbury Press, North Scituate, MA, 1976).
9. R. ADAMS and P. W. McMILLAN, *J. Mater. Sci.* **12** (1977) 643.

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