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Statistical analysis of strength distribution of alumina based single fibres accounting for fibre diameter variations

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The strength distribution of alumina single fibres has been evaluated at room temperature. It has been shown that the classical Weibull distribution function cannot account for the observed dispersions for different gauge lengths. New statistical descriptions have been developed, which take into account the fact that the fibre has an irregular cross-section. Rupture probability functions are derived for cases where the characteristic length for diameter variation is much smaller or much larger than the gauge length. The fibre has then to be viewed as a structure. The material constituting the structure was supposed to obey a simple Weibull law. The method requires the fit of the Weibull parameters of the fibre material and the determination of the geometrical characteristics of the fibres.

1. Introduction

The mechanical properties of continuously reinforced metal or ceramic matrix composites strongly depend on those of the fibres. It is therefore necessary to characterize the mechanical properties of the individual fibres to be able to design the proper composite materials. The purpose of this study was to analyse the room temperature rupture properties of two types of alumina based single fibres (FP and PRD-166, Dupont de Nemours). Ceramics are characterized by variable strength, which is usually attributed to different size flaws preexisting in the material. Rupture strength is therefore a stochastic variable, which is commonly described by a Weibull law. It is actually observed that the Weibull law does not allow correct fitting of the experimental data, so that multimodal Weibull distributions are often used to represent the data. This can be viewed as a way of using more adjustable parameters to fit the data, since it is sometimes difficult to identify defect populations. In this study it was assumed that the material constituting the fibres obeys a simple Weibull law. Discrepancies between this simple model and the experimental data are then attributed to the fact that the fibre is not a perfect cylinder, but actually has a wavy external diameter. Several hypotheses were then tested. It was shown that fibre diameters are not constant, accounting for the observed discrepancy with the classical Weibull model.

2. Experimental procedure

Two fibres were selected for this study: (1) FP alumina fibres, (2) PRD-166 alumina–zirconia fibres (20 wt% ZrO_2). Both fibres are almost fully dense, small cavities are present at grain boundaries. Fibre diameters were measured on the polished section of a bundle embedded in an epoxy resin using image analysis. Seven hundred individual sections were measured for each fibre; the results are given in Table I. Diameter scatter is relatively important. In order to estimate diameter variation along one fibre, several measurements (at 1 mm steps) were made on a 150 mm long sample using scanning electron microscopy (SEM). The relative standard deviation, δ_S, is equal to 3.0% for the FP fibre and to 2.8% for the PRD fibre. This is significantly smaller than the dispersion on a batch of fibres and equal to the dispersion that would result from a surface roughness corresponding to one grain. It is therefore possible to consider that each fibre has a constant diameter, but that the diameter varys from one fibre to another.

Mean grain sizes, G, were measured with a transmission electron microscope (TEM) photographs of ion milled fibres [1], using an automatic image analyser. The results are for FP: $G_{Al_2O_3} = 0.50\ \mu m$; for PRD: $G_{Al_2O_3} = 0.34\ \mu m$, $G_{ZrO_2} = 0.15\ \mu m$.

Tensile tests were performed at room temperature using different gauge lengths (5, 25, 75 and 150 mm) at a constant displacement rate. For each gauge length, 30 individual fibres were tested. This number of tests was required in order to obtain reliable statistical parameters [2]. The experimental setup has been described elsewhere [3, 4].

3. Results and discussion

3.1. Data analysis: classical Weibull approach

The Weibull statistical description of the rupture of brittle materials, which assumes that the rupture of the

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TABLE I Sections of FP and PRD fibres

Fibre	$\bar{S}(\mu m^2)$	$\Lambda(S)\ (\mu m^2)$	$\delta_s(\%)$	$\bar{D}(\mu m)$
FP	269.7	40.8	15.1	18.5
PRD166	242.6	45.7	19.0	17.6

weakest link will result in the failure of the whole structure, states that the rupture probability, P_R, is related to the applied stress, σ, by the following relation

$$P_R(\sigma) = 1 - \exp\left[-\frac{V}{V_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{1}$$

where V is the volume of the structure, V_0 a reference volume, σ_0 a reference stress and m a constant representing data dispersion. The existence of a stress, σ_u, below which failure does not occur is sometimes accounted for. For ceramic materials, it is generally accepted that this stress is close to zero. For fibres having a constant section, the previous equation can be rewritten as

$$P_R(\sigma) = 1 - \exp\left[-\frac{L}{L_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{2}$$

where L is the fibre length and $L_0 = V_0/\bar{S}$. $\bar{S}$ is the average fibre diameter*. Parameters representative of the Weibull distribution, i.e. m and σ_0, can be estimated using two independent methods:

1. a least square fit linear regression for each gauge length, since

$$\ln[-\ln(1-P_R)] = \ln(L/L_0) + m\ln(\sigma) - m\ln(\sigma_0)$$

an alternative method consists in the use of a maximum likelihood method for each gauge length; and

2. using the average value of the rupture stress, which is given by

$$\bar{\sigma} = \sigma_0\left(\frac{L}{L_0}\right)^{-1/m}\Gamma\left(1+\frac{1}{m}\right) \tag{3}$$

Fits using the first method on different gauge lengths, as well as the second method, should give the same values for m and σ_0.

Weibull plots for each gauge length are shown on Figs 1 and 2. Values of m and σ_0 are indicated in Table II for both the least square fit method and the maximum likelihood method. The reference length, L_0, was taken as equal to 5 mm. Mean rupture stresses as a function of the gauge length are plotted on Fig. 3 for both fibres. From this plot it can be deduced, using Equation 3, the following values of m and σ_0

$$\begin{array}{lcc} & m & \sigma_0(\text{MPa}) \\ \text{FP} & 10.84 & 1852 \\ \text{PRD} & 12.03 & 2062 \end{array} \tag{4}$$

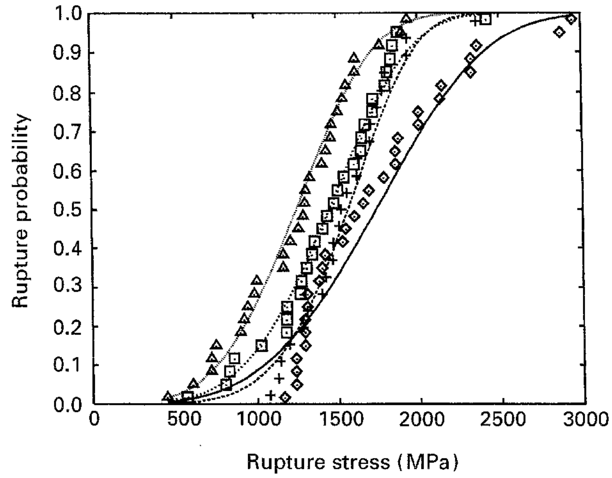

Figure 1 Weibull plots for each gauge length of the FP fibre. Lines indicate fitted curves: (◇) 5 mm, (+) 25 mm, (□) 75 mm, and (△) 150 mm.

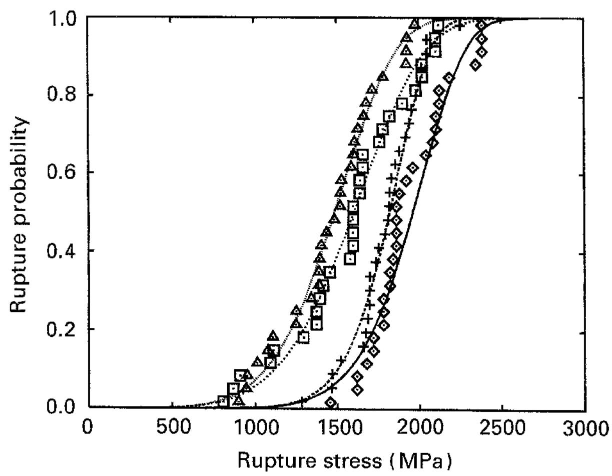

Figure 2 Weibull plots for each gauge length of the PRD fibre. Lines indicate fitted curves: (◇) 5 mm, (+) 25 mm, (□) 75 mm and (△) 150 mm.

Values of m calculated with both methods do not agree. Values obtained by the second method will underestimate the experimental dispersion, but will give correct values of the mean rupture strength. Values obtained by the first method for the longest gauge length will overestimate the mean rupture stress for other gauge lengths; on the other hand, values obtained for the shortest gauge length will underestimate the mean rupture stress. This clearly demonstrates that the Weibull model cannot be applied to the rupture of single fibres. It is also of importance to note that several authors have observed the same trend [5, 6].

3.2. New statistical descriptions

As previously noted, the classical Weibull description of the rupture of brittle solids cannot be applied to the failure of ceramic single fibres. Many authors have explained this discrepancy by the presence of different

*Let x be a stochastic variable, its average value will be denoted $\bar{x}$, its standard deviation $\Lambda(x)$ and its coefficient of variation $\delta_x = \Lambda(x)/\bar{x}$.

TABLE II Values of Weibull parameters m and σ_0 for each gauge length for both fibres ($L_0 = 5$ mm)

Fibre		Gauge length, L				
		5 mm	25 mm	75 mm	150 mm	Average
Least square fit method						
FP						
	m	4.32	6.20	4.06	3.55	4.53
	σ_0, MPa	1912	2163	3090	3590	
PRD						
	m	9.59	10.26	4.74	5.54	7.73
	σ_0, MPa	21039	2206	3051	2930	
Maximum likelihood method						
FP						
	m	3.68	5.24	4.11	3.76	4.20
	σ_0, MPa	1914	2275	3050	3384	
PRD						
	m	8.44	9.76	5.23	5.62	7.26
	σ_0, MPa	2045	2224	2878	2900	

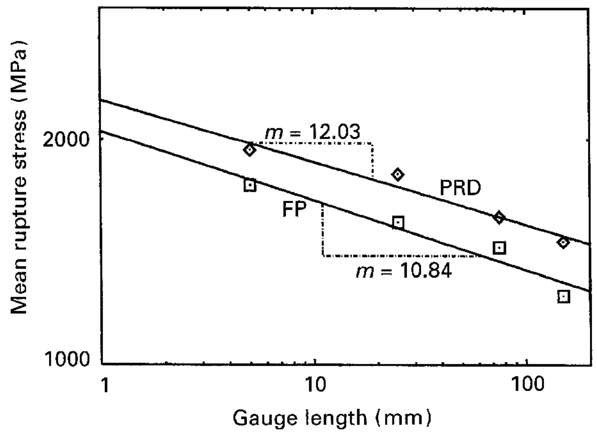

Figure 3 Mean rupture stress versus gauge length for both fibres.

defect populations located either on the surface or in the bulk of the specimen [6–8]. The Weibull equation, (1), is then rewritten as follows

$$P_R(\sigma) = 1 - \exp\left\{ -\frac{V}{V_0} \underbrace{\left[\left(\frac{\sigma}{\sigma_{vi}}\right)^{m_{vi}} + \cdots\right]}_{\text{Volume defect populations}} - \frac{\Sigma}{\Sigma_0} \underbrace{\left[\left(\frac{\sigma}{\sigma_{si}}\right)^{m_{si}} + \cdots\right]}_{\text{Surface defect populations}} \right\} \quad (5)$$

where m_{vi} and σ_{vi} (respectively m_{si} and σ_{si}) represent the Weibull parameters of the ith volume (respectively, surface) defect population. Σ represents the surface of the sample and Σ_0 a reference surface. Using a sufficiently high number of populations will always allow correct representation of the experimental measurements. In the absence of clear microstructural observation of several defect populations, data analysis can be viewed as a curve fitting exercise. In the following discussion, several explanations for the discrepancy between the Weibull model and the ceramic rupture data will be proposed. It will be assumed that the fibre material obeys a simple Weibull law, Equation 1; m and σ_0 being the material Weibull parameters.

3.2.1. Preselection of samples during preparation

Fibres generally have to be extracted from a bundle prior to testing. This can cause the fibre to break thus affecting the statistical analysis of fibre strength: the longer the fibre the higher the load, F_p, necessary to extract it from the bundle. It is assumed that for a given length, F_p is constant. All fibres having a critical defect of $\sigma \leqslant \sigma_p = F_p/S$ will break during sample preparation. The observed rupture probability P_R^* will therefore be equal to

$$P_R^* = 0 \quad \text{if } \sigma \leqslant \sigma_p \int_{\sigma_p}^{\sigma} \left(\frac{\partial P_R}{\partial s} \mathrm{d}s\right) \Big/ \int_{\sigma_p}^{+\infty} \left(\frac{\partial P_R}{\partial s}\right) \mathrm{d}s$$

$$= \frac{P_R(\sigma) - P_R(\sigma_p)}{1 - P_R(\sigma_p)} \qquad \text{if } \sigma > \sigma_p \quad (6)$$

For the short gauge length, i.e. 5 mm, it can be assumed that the preload due to the sample preparation is negligible, so that the Weibull parameters are correctly measured using the first method (see above). For higher gauge lengths, preload is more important. The Weibull plots for $L = 5$ and $L = 150$ mm obtained using method 1 are shown on Fig. 4a, b. The plot for $L = 150$ mm, obtained with the Weibull parameters for $L = 5$ mm, is also indicated, Fig. 4c. It is now assumed that the 150 mm long fibres are subjected to a preload stress equal to 500 MPa, Fig. 4d, or 800 MPa, Fig. 4e; both curves (d) and (e) are calculated using Equation 6. It can clearly be seen from Fig. 4 that preload cannot explain the discrepancy between Weibull's model and the present data.

3.2.2. Effect of section variation: small characteristic length

In this section it is assumed that the fibre diameter varies over a characteristic length, l, much smaller than the fibre length, L, ($l/L \ll 1$). The fibre

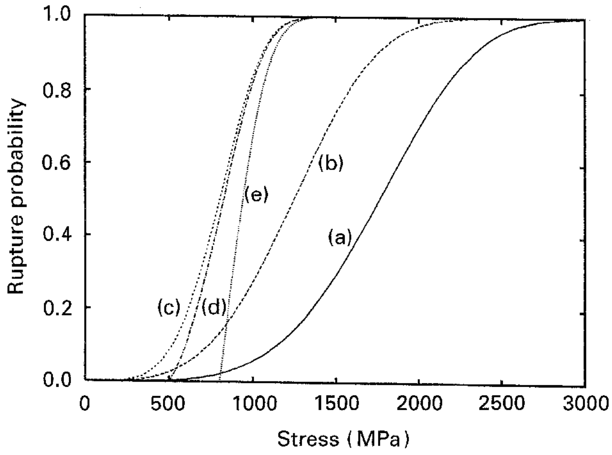

Figure 4 Effect of preload on measured rupture probability (see text): Weibull plot for (a) $L = 5$ mm; (b) $L = 150$ mm; (c) $L = 150$ mm, calculated from data at $L = 5$ mm; (d) Fig. 4c with prestress equal to 500 MPa; and (e) Fig. 4c with prestress equal to 800 MPa.

cross-section is then given by

$$S = \bar{S}[1 + \varepsilon(x/l)]$$

where ε is a function of the fibre co-ordinate x. ε is characterized by the following properties:

1. $\varepsilon \ll 1$, so that notch effects can be neglected,
2. $\bar{\varepsilon} = 0$, and
3. ε is a periodic function (the period is equal to 1).

The non failure probability of a fibre slice of thickness, dx, under load, F, having a section, $S(x)$, is

$$\begin{aligned}\delta P_{\mathrm{NR}}(x) &= \exp\left[-\frac{S\mathrm{d}x}{V_0}\left(\frac{F}{S\sigma_0}\right)^m\right] \\ &= \exp\left\{-\frac{\bar{S}\mathrm{d}x}{V_0}\left(\frac{\sigma^*}{\sigma_0}\right)^m[1+\varepsilon(x/l)]^{1-m}\right\}\end{aligned} \tag{7}$$

with $\sigma^* = F/\bar{S}$. The non-failure probability of the fibre is $P_{\mathrm{NR}} = \prod_{\mathrm{d}x}\delta P_{\mathrm{NR}}(x)$. Therefore

$$\ln(P_{\mathrm{NR}}) = -\frac{\bar{S}}{V_0}\left(\frac{\sigma^*}{\sigma_0}\right)^m\int_0^L[1+\varepsilon(x/l)]^{1-m}\mathrm{d}x \tag{8}$$

Finally, the rupture probability of the fibre is given by

$$P_{\mathrm{R}} = 1 - \exp\left[-\frac{\bar{S}L}{S_0}\left(\frac{\sigma^*}{\sigma_0}\right)^m\chi\right]$$

with

$$\chi = \int_0^1[1+\varepsilon(y)]^{1-m}\mathrm{d}y \tag{9}$$

In the case of multimodal defect distributions it can be shown, using a similar method, that the general Equation 5 has to be rewritten as

$$\begin{aligned}P_{\mathrm{R}} = 1 - \exp\Bigg\{&-\frac{\bar{S}L}{V_0}\left[\cdots+\left(\frac{\sigma^*}{\sigma_{vi}}\right)^{mvi}\chi_{vi}+\cdots\right] \\ &-\frac{(4\pi\bar{S}L)^{1/2}}{\Sigma_0}\left[\cdots+\left(\frac{\sigma^*}{\sigma_{si}}\right)^{msi}\chi_{si}+\cdots\right]\Bigg\}\end{aligned} \tag{10}$$

with

$$\chi_{vi} = \int_0^1[1+\varepsilon(y)]^{1-mvi}\mathrm{d}y$$

and

$$\chi_{si} = \int_0^1[1+\varepsilon(y)]^{1/2-msi}\mathrm{d}y$$

The most important consequence of the previous equation is that the fact of having an irregular fibre does not modify the rupture probability function: if the material rupture stress obeys a Weibull law, the standard rupture stress, σ^*, will also follow a Weibull law, with the same m parameters and slightly different reference stresses (since χ is always close to 1). The results presented in this study cannot therefore be explained by assuming that the fibres have an irregular cross-section.

3.2.3. *Effect of section variation: large characteristic length*

It is now assumed that each fibre has a given section, S, and that the section varies among the different fibres (this is what was experimentally observed on the FP and PRD fibres). The section distribution function is noted as f_S. Since it is practically impossible to measure the diameter of each fibre before testing, the basis for statistical data analysis is the standard rupture stress, σ^*, defined as

$$\sigma^* = \frac{\text{measured rupture force}}{\bar{S}} \tag{11}$$

Weibull parameters m and σ_0 are fitted using σ^* instead of the actual stress results in an increased data dispersion (since sections are dispersed), so that m is underestimated. Let P_{R}^* be the rupture probability associated to σ^*. P_{R}^* is the actual experimentally observed rupture probability; x is the normalized section. Thus

$$x = \frac{S}{\bar{S}} \tag{12}$$

and the distribution function of x, f_x, is given by

$$f_x(x) = \frac{1}{\bar{S}}f_S(S) \tag{13}$$

The reference length, L_0, is defined as follows

$$L = \frac{V_0}{\bar{S}} \tag{14}$$

Considering a fibre of section, S, under load, F, and noting that the stress in the fibre is equal to $\sigma = F/S = \sigma^*/x$, the contribution of this fibre to the rupture probability $\mathrm{d}P_{\mathrm{R}}^*$ is equal to

$$\mathrm{d}P_{\mathrm{R}}^*(\sigma^*) = P_{\mathrm{R}}\left(\frac{\sigma^*}{x}\right)f_x(x)\,\mathrm{d}x \tag{15}$$

S and F are two independent variables, so that the previous equation can be integrated, therefore

$$P_R^*(\sigma^*) = 1 - \int_0^{+\infty} \exp\left[-\frac{L}{L_0} x^{1-m} \times \left(\frac{\sigma^*}{\sigma_0}\right)^m\right] f_x(x)\,dx \quad (16)$$

$P_R^*(\sigma^*)$ can be calculated by numerical integration of the previous formula. The average standard rupture stress can be calculated as follows. Derivation of Equation 16 gives

$$\frac{\partial P_R^*}{\partial \sigma^*} = \int_0^{+\infty} m \frac{L}{L_0} x^{1-m} \frac{\sigma^{*m-1}}{\sigma_0^m} \times \exp\left[-\frac{L}{L_0} x^{1-m}\left(\frac{\sigma^*}{\sigma_0}\right)^m\right] f_x(x)\,dx$$

The average standard rupture stress $\overline{\sigma^*}$ is given by

$$\begin{aligned}\overline{\sigma^*} &= \int_{\sigma^*} \frac{\partial P_R^*}{\partial \sigma^*} \sigma^* \,d\sigma^* \\ &= \int_{\sigma^*}\int_x m \frac{L}{L_0} x^{1-m}\left(\frac{\sigma^*}{\sigma_0}\right)^m \times \exp\left[-\frac{L}{L_0} x^{1-m}\left(\frac{\sigma^*}{\sigma_0}\right)^m\right] f_x(x)\,dx\,d\sigma^* \quad (17) \\ &= \Gamma\left(1+\frac{1}{m}\right)\sigma_0\left(\frac{L_0}{L}\right)^{1/m} \times \int_0^{+\infty} x^{1-1/m} f_x(x)\,dx\end{aligned}$$

since $\int_0^{+\infty} m\alpha^m \exp(-\alpha^m)\,d\alpha = \Gamma(1+\frac{1}{m})$. The following remarks can be made.

1. When f_x is a Dirac function, i.e. there is no section variation, Equations 16 and 17 are equivalent to the classical Weibull distribution, Equations 1 and 3.
2. According to Equation 17, m, can be directly calculated from a linear regression between $\log(\overline{\sigma^*})$ and $\log(L)$, Fig. 3. In addition, m is usually large enough so that $1 - 1/m$ is close to 1 and

$$I_x = \int_0^{+\infty} x^{1-1/m} f_x(x)\,dx \approx 1$$

Therefore σ_0 can be calculated from Equation 17 as

$$\sigma_0 = \overline{\sigma^*}\left(\frac{L}{L_0}\right)^{1/m} \frac{1}{\Gamma(1+1/m) I_x} \quad (18)$$

3. When the material rupture stress is not dispersed, i.e. $m \to +\infty$, Equation 16 can be expressed as

$$P_R^*(\sigma^*) = 1 - \lim_{m\to+\infty} \int_0^{+\infty} \exp\left[-\frac{L}{L_0}\left(\frac{\sigma^*/x}{\sigma_0}\right)^m\right] f_x(x)\,dx \quad (19)$$

The exponential is equal to zero for $\sigma^*/x\sigma_0 > 1$ and equal to 1 for $\sigma^*/x\sigma_0 < 1$. The previous equation can be written as follows

$$\begin{aligned}P_R^*(\sigma^*) &= 1 - \int_{\sigma^*/\sigma_0}^{+\infty} f_x(x)\,dx \\ &= \int_0^{\sigma^*/\sigma_0} f_x(x)\,dx \\ &= F_x\left(\frac{\sigma^*}{\sigma_0}\right) \quad (20)\end{aligned}$$

The standard rupture stress distribution function is therefore equal to the section distribution function.

4. The method can be used for any material strength distribution function, P_R, (a multimodal Weibull function for instance). However, the average standard rupture stress, Equation 17, cannot then be derived analytically, so that it has to be calculated numerically using a proper integration method.

In the case of surface defects the material rupture probability is defined as

$$P_R(\sigma) = 1 - \exp\left[-\frac{\Sigma}{\Sigma_0}\left(\frac{\sigma}{\sigma_0}\right)^{ms}\right] \quad (21)$$

The reference length, L_0, is then defined as $L_0 = \Sigma_0/(4\pi\bar{S})^{1/2}$. It can easily be shown that Equation 16 is rewritten as

$$P_R^*(\sigma^*) = 1 - \int_0^{+\infty} \exp\left[-\frac{L}{L_0} x^{1/2-ms} \times \left(\frac{\sigma^*}{\sigma_0}\right)^{ms}\right] f_x(x)\,dx \quad (22)$$

and the mean rupture stress, Equation 17, is rewritten as

$$\overline{\sigma^*} = \Gamma\left(1+\frac{1}{m_s}\right)\sigma_0\left(\frac{L_0}{L}\right)^{1/ms} \times \int_0^{+\infty} x^{1-1/2ms} f_x(x)\,dx \quad (23)$$

3.3. Application to experimental data

The observed diameter variations for both FP and PRD fibres correspond to section variation over a large characteristic length, so that Section 3.2.3. can be applied. The fibre section repartition function, f_x, is supposed to obey a Gaussian law (average, 1 standard deviation; coefficient of dispersion of the fibre section, Table I). The m and σ_0 parameters were determined using the standard mean rupture stress, Equations 17 and 18, Fig. 3. Equation 17 was integrated using a second-order Runge–Kutta method. Computed and experimental rupture probability for the PRD fibres are shown on Fig. 5, having good agreement. The agreement is not as good for the FP fibre, Fig. 6, but can be improved using a higher fibre section

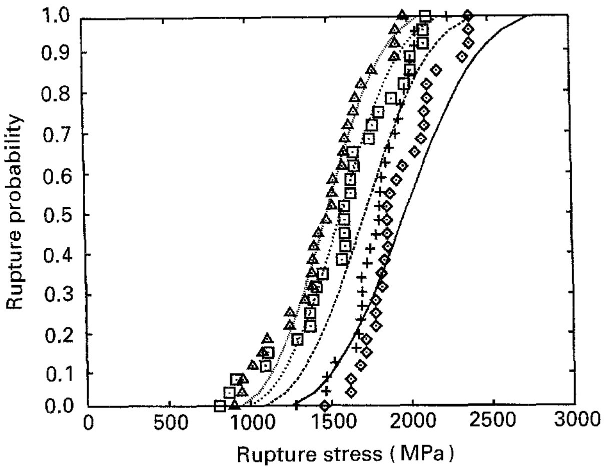

Figure 5 Modified Weibull plot for the PRD fibre, $\delta_x = 19.0\%$. Lines indicate fitted curves: (◇) 5 mm, (+) 25 mm, (□) 75 mm and (△) 150 mm.

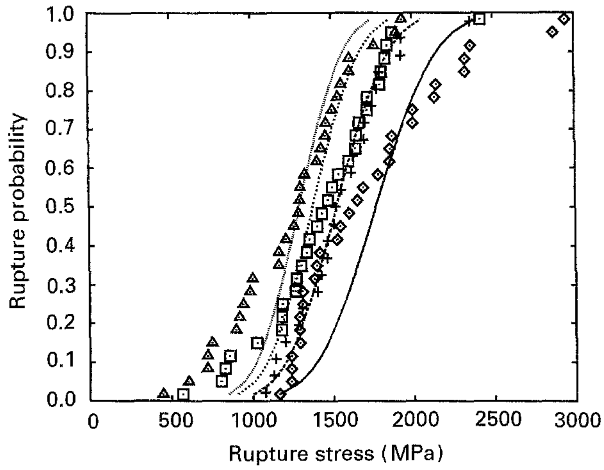

Figure 6 Modified Weibull plot for the FP fibre, $\delta_x = 15.1\%$. Lines indicate fitted curves: (◇) 5 mm, (+) 25 mm, (□) 75 mm and (△) 150 mm.

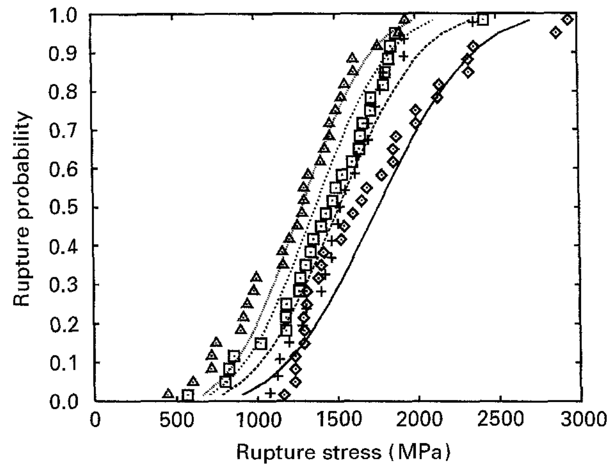

Figure 7 Modified Weibull plot for the FP fibre, $\delta_x = 25.0\%$. Lines indicate fitted curves: (◇) 5 mm, (+) 25 mm, (□) 75 mm and (△) 150 mm.

dispersion coefficient equal to 0.25, Fig. 7. The same computations were carried out assuming a uniform fibre cross-section distribution leading to very similar results. Finally, experimental and computed average rupture stress and standard deviation are compared on Fig. 8, showing good agreement.

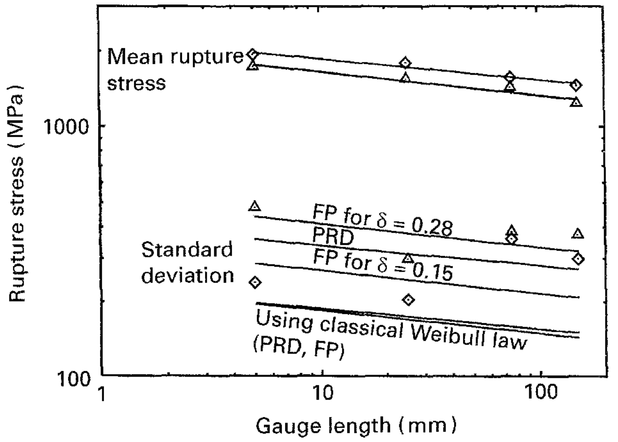

Figure 8 Computed (lines) and experimental (symbols) mean rupture stress and standard deviation for FP (△) and PRD (◇) fibres using the Weibull model modified to account for dispersion on the fibre diameter.

4. Conclusions

Room temperature rupture strength of two alumina based single fibres has been measured for different gauge lengths. It has been shown that the classical Weibull distribution cannot fit the results, as is commonly observed. Using multimodal Weibull laws could indeed allow fitting of the experimental data, but since no evidence for different defect populations was found, other hypotheses based on the geometry of the fibre were investigated, assuming that the material obeys a simple Weibull distribution. This assumption is possibly too simple (in the case of the FP fibre for instance); however, the main topic of this work is to quantify the effect of fibre geometry on the observed rupture statistics. The fibre has therefore to be viewed as a structure. Equations representing the failure probability for preloaded fibres, and fibres having a wavy diameter, have been fully developed. In the case of the studied fibres, it can be considered that the diameter is constant over the gauge length, but that each fibre has a different diameter. The Weibull parameters m and σ_0 are then fitted from the average rupture stress. The rupture probability for each gauge length can then be computed using the fibre diameter distribution function. Results compare well with measured data. It is important to note that the proposed method requires the fit of only two parameters (m and σ_0) and the determination of the diameter distribution function; whereas methods based on multimodal distributions require the fit of more material parameters (at least four), which are sometimes difficult to relate to microstructural features. It is therefore important to account for the fibre geometry when trying to estimate rupture characteristics of single fibres.

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