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A method to distinguish extrinsic and intrinsic fracture-origin populations in monolithic ceramics

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Abstract

Fracture-stress data and fractographic examination for the identification of the fracture origin are presented for two types of brittle materials (tungsten carbide and silicon carbide (SiC)) tested either in three- or four-point bending. The results of the adjustment of a mixture of two Weibull distributions to the fracture-stress data through maximum likelihood estimates via R-software (version 1.8.1 2003, package MASS) agree quite well with the results of the fractographic examinations. Different flaw distributions (e.g. extrinsic and intrinsic) are observed, and each has its own strength distribution parameters.

Keywords: Failure analysis; Mechanical properties; SiC/WC-Co; Wear parts; Maximum likelihood estimation

1. Introduction

Weibull distribution is generally adopted as the underlying distribution of uniaxial strength data of brittle materials under the assumption that specimens are nominally equal (same material and same overall geometry) and fail by the same type of critical flaw. In statistical terminology, this means that sample data are independent and have the same underlying statistical distribution. However, it is also well known that different types of flaws may exist in the specimens, and in these cases the adjustment of a single Weibull distribution is inappropriate. This is recognized by the existing versions of standards for statistical analysis of ceramic strength data.[1,2]

The adequacy of considering a mixture of two Weibull distributions to model the strength of brittle materials was investigated on several fibres, including different types of carbon fibres[3] and SiC fibres.[4] Following the Akaike's criterion[5], the adjustment of a mixture of two Weibull distributions to each type of fibres proved to be more adequate than the adjustment of one single Weibull distribution. However, the model could not be validated through a comparison with the number of specimens that failed due to intrinsic or extrinsic defects because of the difficulties in performing fractographic examination in those 7 μm diameter carbon fibres and 14 μm diameter SiC fibres. Therefore, in the present study, the materials under investigation are bulk ceramics in which the fractographic examination for the identification of the fracture origin was carried out.

As proposed in the current versions of the ISO and CEN standards[1,2], the fracture strength of ceramic specimens with a single population of flaws is a random variable x (>0) that follows a two-parameter Weibull distribution. Since the specimens have the same geometry and are tested under the same mechanical conditions, the specimen volume is not considered and the underlying Weibull distribution is assumed to have the following density function:

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

The likelihood function (L) is the joint density of the random variables and is a function of the unkown parameters (shape m

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and scale σ_0) given the data:[6]

$$L(x_1, \ldots, x_N; m, \sigma_0) = \prod_{i=1}^{N} \left(\frac{m}{\sigma_0}\right)\left(\frac{x_i}{\sigma_0}\right)^{m-1} \exp\left[-\left(\frac{x_i}{\sigma_0}\right)^{m}\right] \quad (2)$$

Assuming a mixture of two Weibull distributions, the cumulative density function is expressed by:

$$f(x) = p\left[\frac{m_1}{\sigma_{01}}\left(\frac{x}{\sigma_{01}}\right)^{m_1-1} \exp\left(-\left(\frac{x}{\sigma_{01}}\right)^{m_1}\right)\right] + (1-p)\left[\frac{m_2}{\sigma_{02}}\left(\frac{x}{\sigma_{02}}\right)^{m_2-1} \exp\left(-\left(\frac{x}{\sigma_{02}}\right)^{m_2}\right)\right] \quad (3)$$

where p is the mixing parameter [0,1] and m_i and σ_{0i} (for $i = 1, 2$) are the two parameters from each single Weibull distribution. The corresponding likelihood is given by:

$$L(x_1, \ldots, x_N; p, m_1, \sigma_{01}, m_2, \sigma_{02}) = \prod_{i=1}^{N}\left\{p\left[\frac{m_1}{\sigma_{01}}\left(\frac{x_i}{\sigma_{01}}\right)^{m_1-1} \exp\left(-\left(\frac{x_i}{\sigma_{01}}\right)^{m_1}\right)\right] + (1-p)\left[\frac{m_2}{\sigma_{02}}\left(\frac{x_i}{\sigma_{02}}\right)^{m_2-1} \exp\left(-\left(\frac{x_i}{\sigma_{02}}\right)^{m_2}\right)\right]\right\} \quad (4)$$

Maximum likelihood (ML), also called the maximum likelihood method, is the procedure of finding the value of one or more parameters (m and σ_0) for a given statistic, which makes the known likelihood distribution a maximum. It is often mathematically easier to manipulate this function by first taking the logarithm of it (log-likelihood function). In this case, parameters are estimated by taking the partial derivatives of the logarithm of the likelihood function ($\ln L$) with respect to the unknown parameters and equating the resulting expressions to zero. The parameter estimates obtained using the ML method are unique (for a two-parameter Weibull distribution), and as the size of the statistical sample increases, the estimates statistically approach the true values of the population more efficiently than other parameter estimation methods. Despite the fact that Weibull parameters are commonly estimated by graphical method through Weibull probability plots which use empirical failure probability, it should be emphasized that the current versions of the ISO and CEN standards[1,2] only recommend the use of ML estimates. The use of other methods of estimating the shape and scale parameters (such as the graphical method or least squares fitting of a straight line to the ranked data points) is not permitted by those standards because they provide less reliable estimates. Therefore, graphical representation of data using a ranking estimator for the probability of failure, e.g. $P_{\mathrm{fi}} = (i - 0.5)/N$ where i is the ranking number once the failure data are ordered from smallest to largest and N is the total number of specimens—should not be used to provide correct assessment of the Weibull distribution parameters, but only to provide visualisation of the distribution of strengths to facilitate some criterion for judging the satisfactory nature of linearity of fit and the detection of one or two low (or high) strength outliers.

In this paper, the shape m and the scale σ_0 parameters of each Weibull distribution – as well as the percentage of data in each distribution when a mixture of two Weibull distributions is assumed – were determined using a ML method implemented at R-software (version 1.8.1 2003, package MASS). In this case, direct optimisation of the log-likelihood is performed with numerical derivatives and the estimated standard errors are taken from the observed information, calculated by a numerical approximation. Using the fracture-stress data and the types of fracture origin detected by fractographic examination of specimens of two different ceramics (tungsten carbide and silicon carbide (SiC)), the purpose of this paper is to evaluate the adequacy of the adjustment of a mixture of two Weibull distributions to fracture-stress data investigating the possible occurrence of two different flaw populations. This will be done by the adjustment of a mixture of two Weibull distributions for the whole data set, as well as, the individual adjustments of Weibull distributions to each subset of data defined according to the type of fracture origin (*intrinsic* versus *extrinsic*). The adequacy is evaluated by comparing the estimates of parameters (the shape and scale parameters) obtained by the mixture of two Weibull distributions with those obtained by the adjustment of individual Weibull distributions. In this evaluation it is admitted that, in the mixed model, m_1 and σ_{01} describe one flaw subpopulation and m_2 and σ_{02} describe the other subpopulation. It also includes the comparison between the estimated fractions of the mixture with the relative occurrence of each type of fracture origin calculated from fractographic examination of specimens.

Since all estimates of Weibull parameters are done by the ML method, graphical representations of data points using a ranking estimator for the probability of failure (and plotting $\ln(1/(1 - P_{\mathrm{fi}}))$ as the ordinate and $\ln \sigma_{\mathrm{fi}}$ as the abscissa) are only used in the present paper for detection of low or high strength outliers.

2. The analysis of tungsten carbide strength data

The material (WC containing 4.5 wt% Co) was supplied by DURIT: Metalurgia Portuguesa do Tungsténio Ltd. in the form of rectangular bars of 20 mm (length) × 6.5 mm (width) × 5.5 mm (height). After sinter-HIP, the bars were ground (average surface roughness, R_a, of 0.02 μm) and the edges were bevelled at 45° using a diamond grinding-wheel, so that chamfers with 0.15 mm width were created. The density of the material (after sintering) measured by the Archimedes' method was 14.93 ± 0.01 g/cm^3. The average grain size of the sintered material was determined by the linear intercept method (Heyn's method) according to the ASTM E112-96 standard, and the average value was 0.41 ± 0.04 μm (ASTM grain size no. 19), which it is typical of a micrograined WC.

Table 1 summarizes the strength data obtained from three-point bend tests conducted on 46 specimens. The distance between the external roller pins was 15.0 mm (according to the ASTM B406-96 standard). All tests were carried out on an Instron electromechanical testing machine using a 5 mm/min crosshead displacement speed.

Fractographic examination (observation of the fracture surfaces), by means of SEM and optical microscopy, in order to identify the fracture origin[7] was carried out at INETI in Lisbon by one of the authors. Only 33 of the 46 tested specimens were examined and only two different types of fracture origin were assigned: *intrinsic* and *extrinsic*. Therefore, the nature of the fracture origin in each specimen was classified in only one of the two types (as shown in Table 1). When the source of failure was located at a chamfer or elsewhere at the outer surface under tension (defects resulting from machining damage), the fracture origin was classified as *extrinsic*. When the source of failure was located at a pore or at other inhomogeneity inside the specimen, the fracture origin was classified as *intrinsic*. For the remainder ($46-33=13$) specimens, which were broken in more than two pieces, the fracture origin is therefore *unknown*. Details about the fractographic examination of the tungsten carbide specimens are described in a previous work.[8]

Table 1
Tungsten carbide observational data

Specimen no.	Fracture stress σ_f (MPa)	Type of fracture origin
1	1710	Extrinsic
2	1467	Extrinsic (at the chamfer)
3	1615	Extrinsic (at the chamfer)
4	1690	Extrinsic (at the chamfer)
5	1049	Extrinsic (at the chamfer)
6	936	Extrinsic (at the chamfer)
7	1392	Unknown
8	1667	Unknown
9	1905	Extrinsic
10	1588	Unknown
11	1254	Extrinsic (at the chamfer?)
12	1160	Extrinsic (at the chamfer)
13	1325	Unknown
14	1110	Intrinsic
15	1637	Unknown
16	1687	Intrinsic
17	1258	Unknown
18	1472	Intrinsic
19	1317	Extrinsic (at the chamfer)
20	1329	Extrinsic
21	1214	Extrinsic (at the chamfer)
22	1861	Unknown
23	1640	Intrinsic
24	1784	Unknown
25	1730	Intrinsic
26	1175	Extrinsic (at the chamfer)
27	1807	Unknown
28	1254	Unknown
29	1558	Extrinsic (at the chamfer)
30	1341	Unknown
31	1076	Extrinsic (at the chamfer)
32	1634	Extrinsic (at the chamfer?)
33	1124	Extrinsic (at the chamfer)
34	1307	Extrinsic (at the chamfer)
35	1567	Intrinsic
36	2298	Extrinsic
37	2004	Extrinsic
38	1924	Extrinsic
39	1547	Extrinsic (at the chamfer)
40	1911	Extrinsic
41	1614	Unknown
42	1733	Intrinsic
43	1759	Intrinsic
44	1756	Intrinsic
45	1681	Intrinsic
46	1510	Unknown

According to Table 1, the summary of the fractographic examination is the following:

- total no. of specimens where the fracture origin is *unknown* = 13;
- total no. of specimens where the fracture origin was *identified* = 33;
- total no. of specimens where the fracture origin is *intrinsic* $N_{\text{int}}=10$;
- total no. of specimens where the fracture origin is *extrinsic* $N_{\text{ext}}=23$ (14–16 of them at the chamfer).

Using the ML method implemented at R-software (version 1.8.1 2003, package MASS), the analysis of tungsten carbide strength data started with the 33 data where the fracture origin was identified (see Table 1). A two-step procedure was used: (1) first of all, only one Weibull distribution was assumed to approximate the true distribution of strengths observed (i.e. the percentage of data is that Weibull distribution was assumed to be $p=100\%$) and the ML estimates of the Weibull parameters ($\hat{m}$ and σ_0) were determined. (2) After that, a mixture of two Weibull distributions (Weibull 1 and Weibull 2) was assumed to model the 33 strength data, and then the ML estimates of % of data in each of the two distributions (p_1 and p_2, respectively; with $p_1+p_2=100\%$), together with the estimates of the parameters of Weibull 1 and Weibull 2, were determined.

In order to use all tungsten carbide strength data that are available in Table 1, the same two-step procedure was followed using those 46 strength data.

The results of the various estimates obtained by the ML method implemented at R-software are shown in Table 2.

A comparison of the ML estimates and the corresponding standard errors obtained from the 33 and 46 data points indicates the following:

(i) The adjustment of one single Weibull distribution to both sets of data confirms that the distribution parameters (shape and scale) are very similar between the two sets of data, with a very small increase in $\hat{m}$ when the data sample size increased from 33 to 46.

(ii) The estimates of the fraction of the mixture of the two Weibull distributions indicates that:

In the 33-data, most of the data ($\approx$81%) follows Weibull 1 distribution with $\hat{m}\approx 5$ and $\sigma_0\approx 1600$ MPa while the remaining data ($\approx$19%) follows Weibull 2 distribution with $\sigma_0\approx 1700$ MPa but with a much higher $\hat{m}$ (47 ± 29).

In the 46-data, the fraction estimate assigned to Weibull 2* distribution ($\approx$16%) is similar to the proportion of the data used to estimate the Weibull 2; the estimates of σ_0 are also similar ($\approx$1700 MPa); and the estimate of the shape parameter $\hat{m}$ (30 ± 16) is also in the range of the high m-value estimated for Weibull 2 when the 33-data is used.

Table 2
Estimates of the Weibull parameters and % of data in each distribution using the WC data

Statistical sample	Assumption for the underlying theoretical distribution	ML estimates		
		% of data in each distribution	Shape $\hat{m}$	Scale σ_0 (MPa)
33-data	One single Weibull distribution		5.3 (±0.7)	1654 (±57)
	Mixture of two Weibull distributions			
	Weibull 1	81 (±11)%	4.7 (±0.7)	1621 (±72)
	Weibull 2	19 (±11)%	47 (±29)	1721 (±27)
46-data	One single Weibull distribution		5.8 (±0.6)	1649 (±44)
	Mixture of two Weibull distributions			
	Weibull 1*	84 (±11) %	5.3 (±0.7)	1629 (±55)
	Weibull 2*	16 (±11)%	30 (±16)	1702 (±43)

Based on these conclusions, and taking into account that the *intrinsic* type of fracture origin was less frequently found by the fractographic examination, we may formulate the following hypothesis: *Weibull 2 and 2* distributions characterize the behaviour of tested specimens in which the fracture origin is intrinsic, while the Weibull 1 and 1* distributions correspond to those where the fracture origin is extrinsic*.

Based on this hypothesis, the number of fracture data in each group was calculated from the above fractions of data that follow Weibull 1 and 1* distributions and Weibull 2 and 2* distributions (Table 3).

Table 3 shows that, in the 33-data set, the ML estimates of fractions of data are in good agreement with the fractographic examination in which $N_{\text{ext}} = 23$ and $N_{\text{int}} = 10$. This result gives support to the separation based on optimisation of the log-likelihood despite the fact that the observed values (N_{ext} and N_{int}) are, respectively, at the lower and the upper bounds of the corresponding estimates ($\hat{N}_{\text{ext}}$ and $\hat{N}_{\text{int}}$).

In order to proceed with the verification of the validity of the hypothesis, we have carried out new ML estimates of one single Weibull distribution adjusted to the 23-data corresponding to the specimens where the fracture origin was identified as *extrinsic*, and we made the same for the 10-data corresponding to the specimens where the fracture origin was identified as *intrinsic*.

For the 23 data where the fracture origin was identified as *extrinsic*, the ML adjustment of one Weibull distribution gives:

$$\text{Shape } \hat{m} = 4.5\,(\pm 0.7),\ \text{scale } \sigma_0 = 1627\,(\pm 79)\text{MPa}$$

For the 10 data where the fracture origin was identified as *intrinsic*, the ML adjustment of one Weibull distribution gives:

$$\text{Shape } \hat{m} = 14.5\,(\pm 4.1),\ \text{scale } \sigma_0 = 1683\,(\pm 38)\text{MPa}$$

However, according to the present versions of ISO or EN standards for statistical analysis of ceramic strength data,[1,2] the estimated $\hat{m}$ values should be corrected using an unbiasing factor b (tabulated in annex to the ISO/DIS 20501 or ENV 843-5 standard):

$$\hat{m}_{\text{corr}} = \hat{m}b$$

Table 4 summarizes the values of σ_0 and $\hat{m}_{\text{corr}}$ for all maximum likelihood estimates. To facilitate the analysis of the results, dedicated graphs are also presented in Figs. 1 and 2.

From the analysis of Figs. 1 and 2 we can conclude that, in fact, the shape and scale parameters of Weibull 1 and 1* distributions are very similar to the parameters of the data where the fracture origin is *extrinsic*; and also the shape and scale param-

Table 3
Estimates of N_{ext} and N_{int} using the WC data

Statistical sample	$\hat{N}_{\text{ext}}$	$\hat{N}_{\text{int}}$
33-data	From 23 to 30	From 3 to 10
46-data	From 34 to 42	From 4 to 12

Table 4
Summary of σ_0 and $\hat{m}_{\text{corr}}$ values for all maximum likelihood estimates using the WC data

Statistical sample	σ_0 (MPa)	$\hat{m}$	$\hat{m}_{\text{corr}}$
Weibull 1* in 46-data	1629 (±55)	5.3 (±0.7)	5.1 (±0.67)
Weibull 1 in 33-data	1621 (±72)	4.7 (±0.7)	4.5 (±0.66)
Extrinsic 23-data	1627 (±79)	4.5 (±0.7)	4.2 (±0.66)
Weibull 2* in 46-data	1702 (±43)	30 (±16)	24 (±13)
Weibull 2 in 33-data	1721 (±27)	47 (±29)	35 (±22)
Intrinsic 10-data	1683 (±38)	14.5 (±4.1)	12.5 (±3.5)
Intrinsic 9-data[a]	1708 (±22)	26.7 (±7.6)	22.5 (±6.4)

[a] Data screening of the fracture-stress values (in MPa) of the 10 specimens where the fracture origin was identified as *intrinsic* (1110, 1687, 1472, 1640, 1730, 1567, 1733, 1759, 1756, 1681) shows that 1110 can be considered as a low strength outlier and therefore this value was discarded.

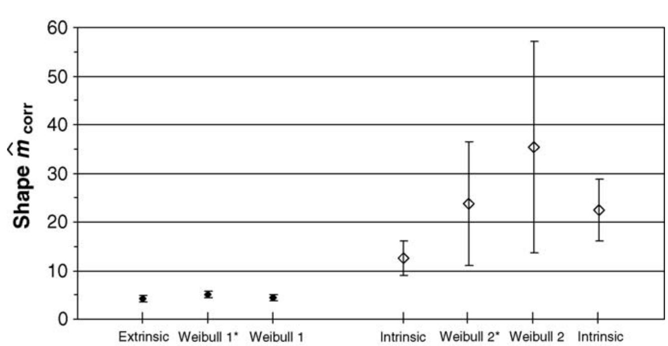

Fig. 1. Comparison of ML estimates of shape parameter ($\hat{m}_{\text{corr}}$) using the WC data.

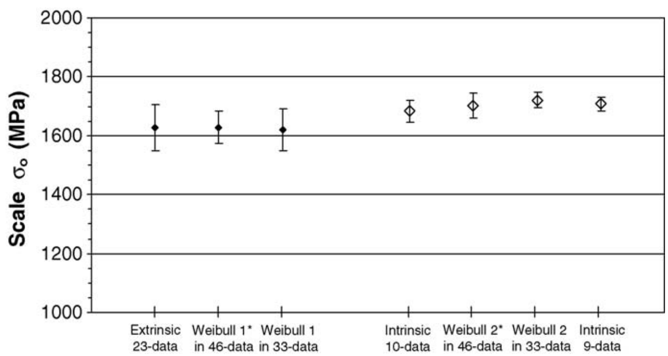

Fig. 2. Comparison of ML estimates of scale parameter (σ_0) using the WC data.

eters of the Weibull 2 and 2* distributions are within the range of the corresponding parameters of the data where the fracture origin is *intrinsic*.

If the 23 extrinsic data is screened for outlying observations (using the traditional graphical representation in a Weibull plot) we do not observe any values appearing as clear outliers (see Fig. 3). However, if we screen the 10 intrinsic data (Fig. 4), one of the observed values (1110 MPa in specimen no. 14) appears as a low strength outlier. This value can be discarded because, although the fractographic examination has assigned it to the intrinsic population, there are some doubts that the real origin of the fracture was intrinsic. In fact, the low strength value of specimen no. 14 is much better housed within the low strength range of the extrinsic population. In Table 4, the statistical sample named "Intrinsic 9-data" consists of the remaining nine observed values.

Therefore, instead of $N_{\text{int}} = 10$ in the aforementioned "33 data where the fracture origin was identified", it will be more adequate to consider that we have $N_{\text{int}} = 9$ and $N_{\text{ext}} = 23$.

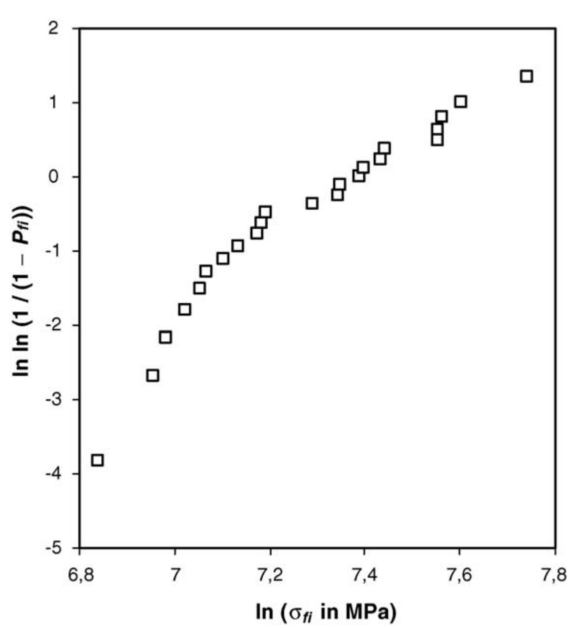

Fig. 3. Weibull plot of the 23 WC extrinsic data using the ranking estimator $P_{\text{fi}} = (i - 0.5)/N$.

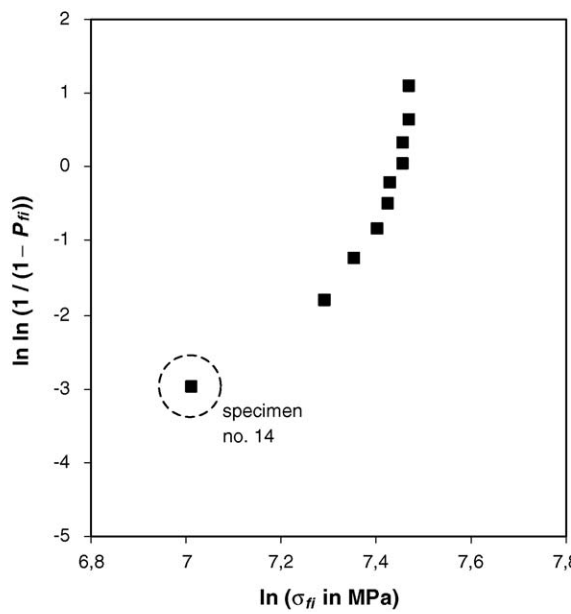

Fig. 4. Weibull plot of the 10 WC intrinsic data using the ranking estimator $P_{\text{fi}} = (i - 0.5)/N$.

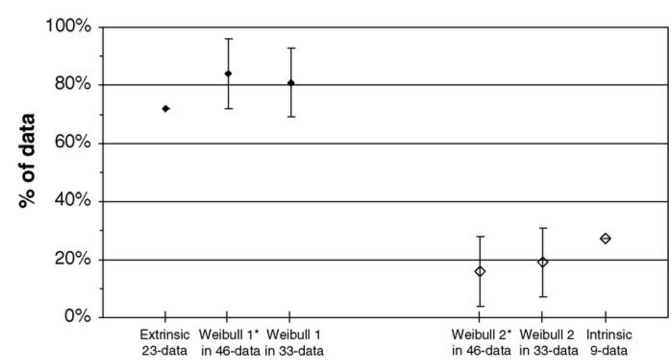

Fig. 5. Comparison between the % of *extrinsic* and *intrinsic* data observed and the % of data belonging to Weibull 1, 1*, 2 and 2* distributions (WC data).

A final comparison of the % of *extrinsic* and *intrinsic* data observed and the % of data belonging to Weibull 1, 1*, 2 and 2* distributions is presented in Fig. 5.

3. The analysis of silicon carbide strength data

The silicon carbide (SiC) material was supplied by ESK (GmbH) in the form of plates (48 mm × 48 mm × 12 mm). According to the supplier, the density of the material is 3.10 g/cm^3 and the porosity is less than 3.5%. Rectangular bars of 45 mm × 4 mm × 4 mm were cut and prepared using conventional procedure and sawing machine. The bars were polished and the edges were chamfered. The specimens were tested in symmetrical four-point bending. The four-point flexure jig had an inner span of 20 mm, and an outer span of 40 mm. The tests were carried out on a conventional Zwick testing machine, using a 1 mm/min crosshead displacement rate.

The strength data obtained in 69 specimens are summarized in Table 5. The fractographic examination to identify the fracture origin was carried out in France exclusively by SEM. The

Table 5
Silicon carbide observational data

Specimen no.	Fracture stress σ_f (MPa)	Location of fracture origin (detected inhomogeneity)
1	306	SS (big pore)
2	368	SS (pore)
3	380	SS (pore)
4	349	SS (big pore)
5	300	SS (big pore)
6	314	S (pore)
7	305	S*
8	387	V (inclusion)
9	410	V (pore)
10	358	V (pore)
11	306	SS (pore)
12	332	V (pore)
13	305	S (pore)
14	364	V (pore)
15	274	C*
16	260	SS (pore)
17	369	V (pore)
18	355	SS (pore)
19	372	V (pore)
20	351	S (pore)
21	384	C (pore)
22	313	SS (pore)
23	362	V (pore)
24	282	C*
25	311	S*
26	392	S (pore)
27	360	S (pore)
28	393	SS (pore)
29	354	S (pore)
30	328	S (pore)
31	358	V (inclusion)
32	383	V (pore)
33	429	SS (pore)
34	308	SS (pore)
35	362	V (pore)
36	256	C*
37	365	V (pore)
38	295	SS (pore)
39	348	SS (pore)
40	265	S*
41	333	S (pore)
42	382	V (pore)
43	352	C*
44	352	V (pore)
45	371	S (pore)
46	276	S*
47	394	SS (inclusion)
48	347	SS (big pore)
49	343	SS (big pore)
50	326	SS (big pore)
51	385	SS (inclusion)
52	355	SS (pore)
53	349	S (pore)
54	314	S*
55	370	S (pore)
56	312	SS (pore)
57	339	S (pore)
58	373	S (pore)
59	372	SS (pore)
60	358	S (pore)
61	355	SS (pore)
62	402	SS (pore)
63	371	SS (pore)
64	343	SS (pore)
65	372	SS (pore)
66	393	V (pore)
67	307	C*
68	292	S*
69	389	SS (pore)

examiner has identified the fracture origin in each specimen and classified it according to its location and type of inhomogeneity detected. Four main types were used for the location of the fracture origin: (1) located at a corner (C) of the specimen; (2) located at the bottom surface (S); (3) located close to the bottom surface, but sub-superficial (SS); and (4) located clearly inside (V). The material inhomogeneity responsible for the failure of the specimen is also mentioned in Table 5 (in brackets). When the fracture origin could not be assigned to an inhomogeneity (e.g. pore or inclusion) the location symbol is followed by an asterisk.

Since in this case the fractography examiner did not make any clear distinction between *intrinsic* and *extrinsic* fracture, we assumed that the specimens in which the fracture origin is not assigned to an inhomogeneity (pore or inclusion) failed by a flaw resulting from surface preparation. This means that the detection of a material inhomogeneity was considered more important than the location (C, S, SS or V) of the fracture origin. Thus, only the data marked with (*) in Table 5 were considered as *extrinsic*.

With the above mentioned assumption, the summary of the fractographic examination becomes the following:

- total no. of specimens where the fracture origin is *unknown* = 0;
- total no. of specimens where the fracture origin was *identified* = 69;
- total no. of specimens where the fracture origin is *intrinsic* $N_{\rm int} = 58$;
- total no. of specimens where the fracture origin is *extrinsic* $N_{\rm ext} = 11$.

A statistical analysis similar to that used in Section 2 for the WC specimens was applied to the 69 SiC fracture-stress observational data. The results of the ML estimates of the Weibull parameters and % of data in each distribution are shown in Table 6.

Based on the results of ML estimates of the % of data in each distribution, and knowing from the fractographic examination that the in the SiC data the *intrinsic* type of fracture origin is more frequent than the *extrinsic* type, the following hypothesis can be formulated: "*Weibull 1 distribution characterizes the behaviour of tested specimens in which the fracture origin is intrinsic, while the Weibull 2 distribution corresponds to those where the fracture origin is extrinsic*". Then, according to this

Table 6
Estimates of the Weibull parameters and % of data in each distribution using the SiC data

Statistical sample	Assumption for the underlying theoretical distribution	ML estimates		
		% of data in each distribution	Shape $\hat{m}$	Scale σ_0 (MPa)
69-data	One single Weibull distribution		10.7 (±1.0)	362 (± 4)
	Mixture of two Weibull distributions:			
	Weibull 1	82.5 (±9)%	13.1 (±2.2)	370 (±6)
	Weibull 2	17.5 (±9)%	21.6 (±9.4)	303 (±6)

Table 7
Estimates of N_{int} and N_{ext} using the SiC data

Statistical sample	$\hat{N}_{int}$	$\hat{N}_{ext}$
69-data	From 51 to 63	From 6 to 18

hypothesis, the number of fracture data in each group was calculated from the fractions of data that follow Weibull 1 and Weibull 2 distributions (Table 7).

The comparison between the ML estimates of fraction ($\hat{N}_{int}$ and $\hat{N}_{ext}$) and the assessed values obtained by the fractographic examination (in which $N_{int} = 58$ and $N_{ext} = 11$) shows that the proportions of each group are within the estimated range, and consequently support the formulated hypothesis.

To proceed with the verification of the validity of the hypothesis, we have carried out new ML estimates of one single Weibull distribution adjusted to the 11-data corresponding to the specimens where the fracture origin was identified as *extrinsic*, and we made the same for the 58-data corresponding to the specimens where the fracture origin was identified as *intrinsic*. The ML estimates are summarized in Table 8 and in the diagrams presented in Figs. 6–8.

Figs. 9 and 10 show the Weibull plots for the 58 intrinsic data and the 11 extrinsic data, respectively. In the 58 intrinsic data (Fig. 9), we do not observe any clear outlier. However, data screening of the fracture-stress values of the 11 specimens where the fracture origin was identified as *extrinsic* shows that the value found for specimen no. 43 (352 MPa) may be considered as a high strength outlier (raising some doubts about the real origin of the fracture, because this high strength value may be assigned to the high strength range of the intrinsic population). Therefore, we have also carried out ML estimates of one single Weibull distribution adjusted to the 10 remaining extrinsic data (see in Table 8 the statistical sample named "Extrinsic 10-data").

Table 8
Summary of σ_0 and $\hat{m}_{corr}$ values for all maximum likelihood estimates using the SiC data

Statistical sample	σ_0 (MPa)	$\hat{m}$	$\hat{m}$
Weibull 1 in 69-data	370 (±6)	13.1 (±2.2)	12.8 (±2.15)
Intrinsic 58-data	369 (±4)	12.9 (±1.3)	12.6 (±1.27)
Weibull 2 in 69-data	303 (±6)	21.6 (±9.4)	19.1 (±8.3)
Extrinsic 11-data	306 (±9)	11.0 (±2.4)	9.6 (±2.1)
Extrinsic 10-data[a]	297 (±6)	17.5 (±4.5)	15.0 (±3.9)

[a] Data screening (see Fig. 10) of the fracture-stress values (in MPa) of the 11 specimens where the fracture origin was identified as *extrinsic* (305, 274, 282, 311, 256, 265, 352, 276, 314, 307, 292) shows that 352 (specimen no. 43) is probably a high strength outlier and therefore this value was discarded.

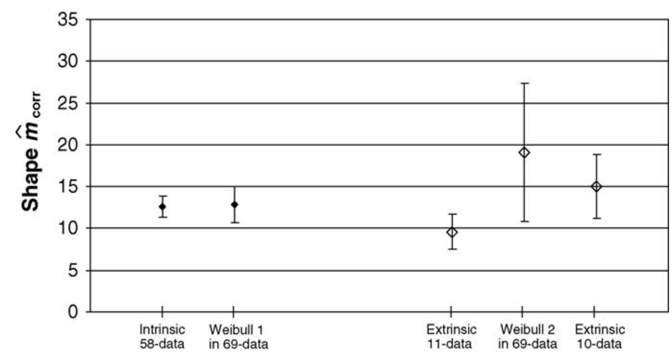

Fig. 6. Comparison of ML estimates of shape parameter ($\hat{m}_{corr}$) using the SiC data.

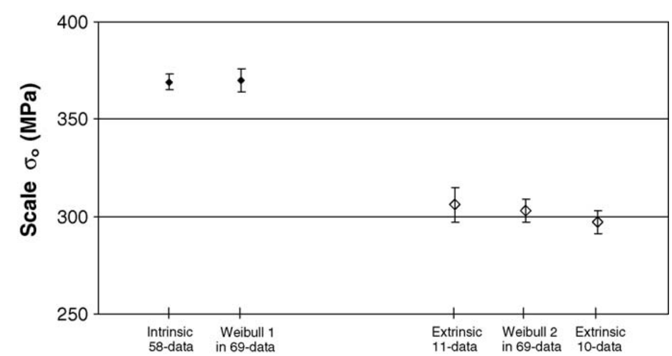

Fig. 7. Comparison of ML estimates of scale parameter (σ_0) using the SiC data.

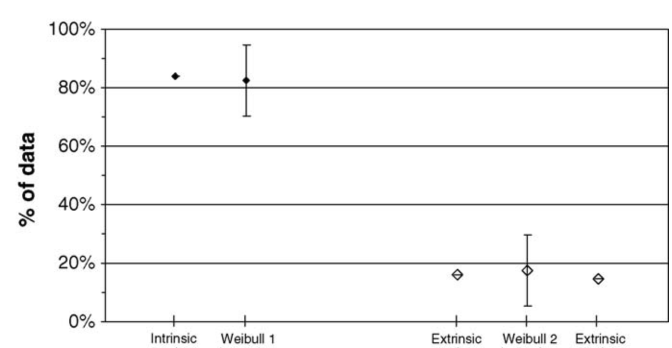

Fig. 8. Comparison between the % of *intrinsic* and *extrinsic* data observed and the % of data belonging to Weibull 1 and 2 distributions (SiC data).

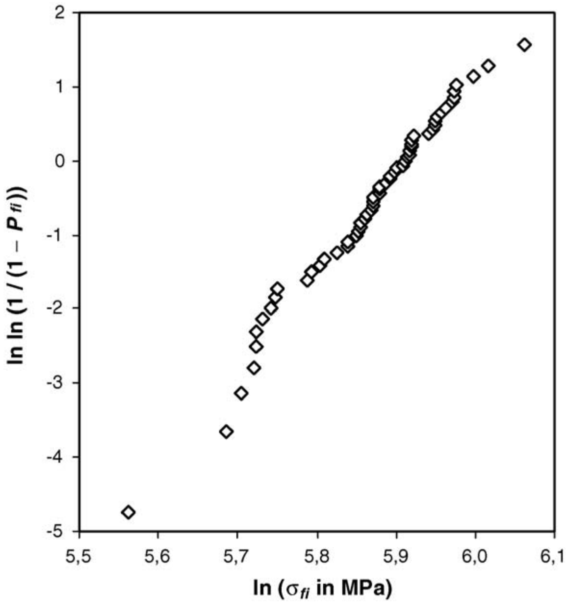

Fig. 9. Weibull plot of the 58 SiC intrinsic data using the ranking estimator $P_{fi} = (i - 0.5)/N$.

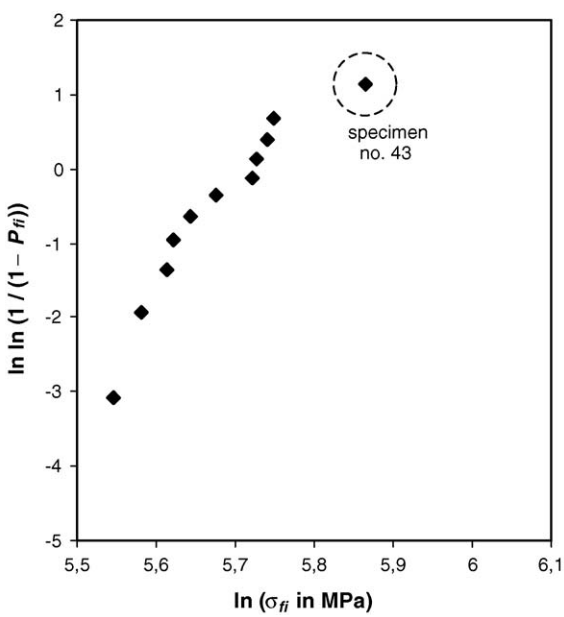

Fig. 10. Weibull plot of the 11 SiC extrinsic data using the ranking estimator $P_{fi} = (i - 0.5)/N$.

4. Concluding remarks

For the tungsten carbide strength data obtained by three-point bend tests conducted in 46 specimens, only a few results of fracture stress were found to represent the intrinsic behaviour of the material. The majority of the fractures are due to extrinsic defects resulting from machining damage and especially from critical flaws in the vicinity of the machined chamfers. The results of the adjustment of a mixture of two Weibull distributions to the fracture-stress data using the maximum likelihood method agree quite well with the results of the fractographic examination. The range of fracture strength values observed in specimens that failed by an extrinsic defect was found to be from 936 up to 2298 MPa, whereas for the specimens that failed by an intrinsic defect the range goes only from 1472 to 1759 MPa (if the 1110 MPa value is considered a low strength outlier and discarded from this population).

The analysis of the strength data obtained by four-point bend tests conducted in 69 specimens of silicon carbide shows also that the adjustment of a mixture of two Weibull distributions to the fracture-stress data is adequate and the maximum likelihood estimates agree quite well with the results of the fractographic examination. Contrary to the WC specimens, the majority of the SiC specimens failed due to the herein called *intrinsic* defects (pores and inclusions) and only a few results of fracture stress were found to be due to *extrinsic* defects of the SiC test pieces which are flaws clearly located either at the bottom surface (S^*) or at one of the edges (C^*) of the specimens. In the case of the silicon carbide data, the range of fracture strength values observed in the specimens that failed by an intrinsic defect was found to be from 260 up to 429 MPa, while for the specimens that failed by an extrinsic defect the values fluctuate from 256 till 352 MPa.

It was therefore demonstrated that maximum likelihood adjustment of a mixture of two Weibull distributions to the fracture-stress data of WC and SiC specimens allowed the identification of two populations of critical flaws (the so-called *extrinsic* and *intrinsic* flaws). The parameter estimates (shape and scale of each Weibull distribution and the percentage of data in each distribution) are in agreement with the parameter values obtained by the adjustment of single Weibull distribution to subset of data defined according to their fracture origin and based on fractographic examinations.

The percentage of data in each population (*intrinsic* versus *extrinsic*) depends on the manufacturing process and on the surface preparation of the test pieces.

This work shows that, in both ceramic materials (WC and SiC), two concurrent populations of critical flaws can be considered (the intrinsic and the extrinsic flaws). Each specimen has intrinsic and extrinsic flaws; but some specimens fail by an intrinsic flaw, while others fail by an extrinsic flaw. The range of fracture strength values of the extrinsic critical flaws is not disjointed from the range of fracture strength values of the intrinsic critical flaws, but both ranges overlap.

Even without knowing a priori the fracture origin of specimens under analysis, the adjustment of a mixture of two Weibull distributions proved to be an adequate tool for the characterization of the fracture behaviour, allowing the perception of the relative occurrence of different types of fracture origin.

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