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Standard Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Graphites[1]

This standard is issued under the fixed designation D7846; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This practice covers the reporting of uniaxial strength data for graphite and the estimation of probability distribution parameters for both censored and uncensored data. The failure strength of graphite materials is treated as a continuous random variable. Typically, a number of test specimens are failed in accordance with the following standards: Test Methods C565, C651, C695, C749, Practice C781 or Guide D7775. The load at which each specimen fails is recorded. The resulting failure stresses are used to obtain parameter estimates associated with the underlying population distribution. This practice is limited to failure strengths that can be characterized by the two-parameter Weibull distribution. Furthermore, this practice is restricted to test specimens (primarily tensile and flexural) that are primarily subjected to uniaxial stress states.

1.2 Measurements of the strength at failure are taken for various reasons: a comparison of the relative quality of two materials, the prediction of the probability of failure for a structure of interest, or to establish limit loads in an application. This practice provides a procedure for estimating the distribution parameters that are needed for estimating load limits for a particular level of probability of failure.

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C565 Test Methods for Tension Testing of Carbon and Graphite Mechanical Materials

C651 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature

C695 Test Method for Compressive Strength of Carbon and Graphite

C749 Test Method for Tensile Stress-Strain of Carbon and Graphite

C781 Practice for Testing Graphite Materials for Gas-Cooled Nuclear Reactor Components

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D7775 Guide for Measurements on Small Graphite Specimens

E6 Terminology Relating to Methods of Mechanical Testing

E178 Practice for Dealing With Outlying Observations

E456 Terminology Relating to Quality and Statistics

3. Terminology

3.1 Proper use of the following terms and equations will alleviate misunderstanding in the presentation of data and in the calculation of strength distribution parameters.

3.2 *Definitions:*

3.2.1 *estimator, n*—a well-defined function that is dependent on the observations in a sample. The resulting value for a given sample may be an estimate of a distribution parameter (a point estimate) associated with the underlying population. The arithmetic average of a sample is, for example, an estimator of the distribution mean.

3.2.2 *population, n*—the totality of valid observations (performed in a manner that is compliant with the appropriate test standards) about which inferences are made.

3.2.3 *population mean, n*—the average of all potential measurements in a given population weighted by their relative frequencies in the population.

3.2.4 *probability density function, n*—the function $f(x)$ is a probability density function for the continuous random variable X if:

$$f(x) \geq 0 \tag{1}$$

and

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard**

$$\int_{-\infty}^{\infty} f(x)\, dx = 1 \quad (2)$$

The probability that the random variable X assumes a value between a and b is given by:

$$\Pr(a < X < b) = \int_{a}^{b} f(x)\, dx \quad (3)$$

3.2.5 *sample, n*—a collection of measurements or observations taken from a specified population.

3.2.6 *skewness, n*—a term relating to the asymmetry of a probability density function. The distribution of failure strength for graphite is not symmetric with respect to the maximum value of the distribution function; one tail is longer than the other.

3.2.7 *statistical bias, n*—inherent to most estimates, this is a type of consistent numerical offset in an estimate relative to the true underlying value. The magnitude of the bias error typically decreases as the sample size increases.

3.2.8 *unbiased estimator, n*—an estimator that has been corrected for statistical bias error.

3.2.9 *Weibull distribution, n*—the continuous random variable X has a two-parameter Weibull distribution if the probability density function is given by:

$$f(x) = \left(\frac{m}{S_c}\right)\left(\frac{x}{\beta}\right)^{m-1} \exp\left[-\left(\frac{x}{\beta}\right)^{m}\right]; \quad x>0 \quad (4)$$

$$f(x) = 0;\ x \le 0$$

and the cumulative distribution function is given by:

$$F(x) = 1 - \exp\left[-\left(\frac{x}{\beta}\right)^{m}\right]; \quad x>0 \quad (5)$$

or

$$F(x) = 0; \quad x \le 0$$

where:

m = Weibull modulus (or the shape parameter) ($m > 0$), and
β = scale parameter (> 0).

3.2.9.1 *Discussion*—The random variable representing uniaxial tensile strength of graphite will assume only positive values, and the distribution is asymmetrical about the population mean. These characteristics rule out the use of the normal distribution (as well as others) and favor the use of the Weibull and similar skewed distributions. If the random variable representing uniaxial tensile strength of a graphite is characterized by Eq 4, and Eq 5, then the probability that the tested graphite will fail under an applied uniaxial tensile stress, σ, is given by the cumulative distribution function:

$$P_f = 1 - \exp\left[-\left(\frac{\sigma}{S_c}\right)^{m}\right]; \quad \sigma>0 \quad (6)$$

and

$$P_f = 0; \quad \sigma \le 0$$

where:

P_f = the probability of failure, and
S_c = the Weibull characteristic strength.

3.2.9.2 *Discussion*—The Weibull characteristic strength depends on the uniaxial test specimen (tensile, compression and flexural) and may change with specimen geometry. In addition, the Weibull characteristic strength has units of stress and should be reported using units of MPa or GPa.

3.3 For definitions of other statistical terms, terms related to mechanical testing, and terms related to graphite used in this practice, refer to Terminologies D4175, E6, and E456, or to appropriate textbooks on statistics (**1-5**).[3]

3.4 *Nomenclature:*

$F(x)$ = cumulative distribution function
$f(x)$ = probability density function
$\mathcal{L}$ = likelihood function
m = Weibull modulus
$\hat{m}$ = estimate of the Weibull modulus
$\hat{m}_U$ = unbiased estimate of the Weibull modulus
N = number of specimens in a sample
P_f = probability of failure
t = intermediate quantity used in calculation of confidence bounds
X = random variable
x = realization of a random variable X
β = Weibull scale parameter
$\hat{\mu}$ = estimate of mean strength
σ = uniaxial tensile stress
σ_i = maximum stress in the ith test specimen at failure
S_c = Weibull characteristic strength (associated with a test specimen)
$\hat{S}_c$ = estimate of the Weibull characteristic strength

4. Summary of Practice

4.1 This practice provides a procedure to estimate Weibull distribution parameters from failure data for graphite data tested in accordance with applicable ASTM test standards. The procedure consists of computing estimates of the biased Weibull modulus and Weibull characteristic strength. If necessary, compute an estimate of the mean strength. If the sample of failure strength data is uncensored, compute an unbiased estimate of the Weibull modulus, and compute confidence bounds for both the estimated Weibull modulus and the estimated Weibull characteristic strength. Finally, prepare a graphical representation of the failure data along with a test report.

5. Significance and Use

5.1 Two- and three-parameter formulations exist for the Weibull distribution. This practice is restricted to the two-parameter formulation. An objective of this practice is to obtain point estimates of the unknown Weibull distribution parameters by using well-defined functions that incorporate the failure data. These functions are referred to as estimators. It is desirable that an estimator be consistent and efficient. In addition, the estimator should produce unique, unbiased estimates of the distribution parameters (**6**). Different types of

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.

estimators exist, such as moment estimators, least-squares estimators, and maximum likelihood estimators. This practice details the use of maximum likelihood estimators.

5.2 Tensile and flexural specimens are the most commonly used test configurations for graphite. The observed strength values depend on specimen size and test geometry. Tensile and flexural test specimen failure data for a nearly isotropic graphite **(7)** is depicted in Fig. 1. Since the failure data for a graphite material can be dependent on the test specimen geometry, Weibull distribution parameter estimates (m, S_c) shall be computed for a given specimen geometry.

5.3 The bias and uncertainty of Weibull parameters depend on the total number of test specimens. Variability in parameter estimates decreases exponentially as more specimens are collected. However, a point of diminishing returns is reached where the cost of performing additional strength tests may not be justified. This suggests a limit to the number of test specimens for determining Weibull parameters to obtain a desired level of confidence associated with a parameter estimate. The number of specimens needed depends on the precision required in the resulting parameter estimate or in the resulting confidence bounds. Details relating to the computation of confidence bounds (directly related to the precision of the estimate) are presented in 8.3 and 8.4.

6. Outlying Observations

6.1 In this context, an outlying observation (outlier) is one that deviates noticeably from other observations in the sample and is an extreme manifestation of the variability of the strength due to non-homogeneity of graphite material, or large disparate flaws, given the prescribed experimental procedure has been followed. Before computing the parameter estimates the data should be screened for outliers. Apparent outliers must be retained and treated as any other observation in the failure sample, e.g. all test results must be included in the computation of the parameter estimates. Only where the outlying observation is the result of a known gross deviation from the prescribed experimental procedure, or a known error in calculating or recording the numerical value of the data point in question, may the outlying observation be censored. In such a case, the test report should record the justification. If a test specimen is deemed unsuitable either for testing, or fails before the prescribed experimental procedure has commenced, then this should not be regarded as a test result. The null test should be fully documented in the test report. The procedures for dealing with outlying observations are detailed in Practice E178.

7. Maximum Likelihood Parameter Estimators

7.1 The likelihood function for the two-parameter Weibull distribution of a censored sample is defined by Eq 7:

$$\mathscr{L} = \prod_{i=1}^{r} \left(\frac{\hat{m}}{\hat{S}_c}\right)\left(\frac{\sigma_i}{\hat{S}_c}\right)^{\hat{m}-1} \exp\left[-\left(\frac{\sigma_i}{\hat{S}_c}\right)^{\hat{m}}\right] * \prod_{j=r+1}^{N} \exp\left[-\left(\frac{\sigma_j}{\hat{S}_c}\right)^{\hat{m}}\right]. \quad (7)$$

7.1.1 For graphite material, this expression is applied to a sample where outlying observations are identified. When Eq 7 is used to estimate the parameters associated with a strength distribution containing outliers, then r is the number of data points retained in the sample, that is, data points not considered outliers, and i is the associated index in the first product. In this practice, the second product is carried out for the outlying observations. Therefore, the second product is carried out from ($j = r + 1$) to N (the total number of specimens) where j is the index in the second product. Accordingly, σ_i is the maximum

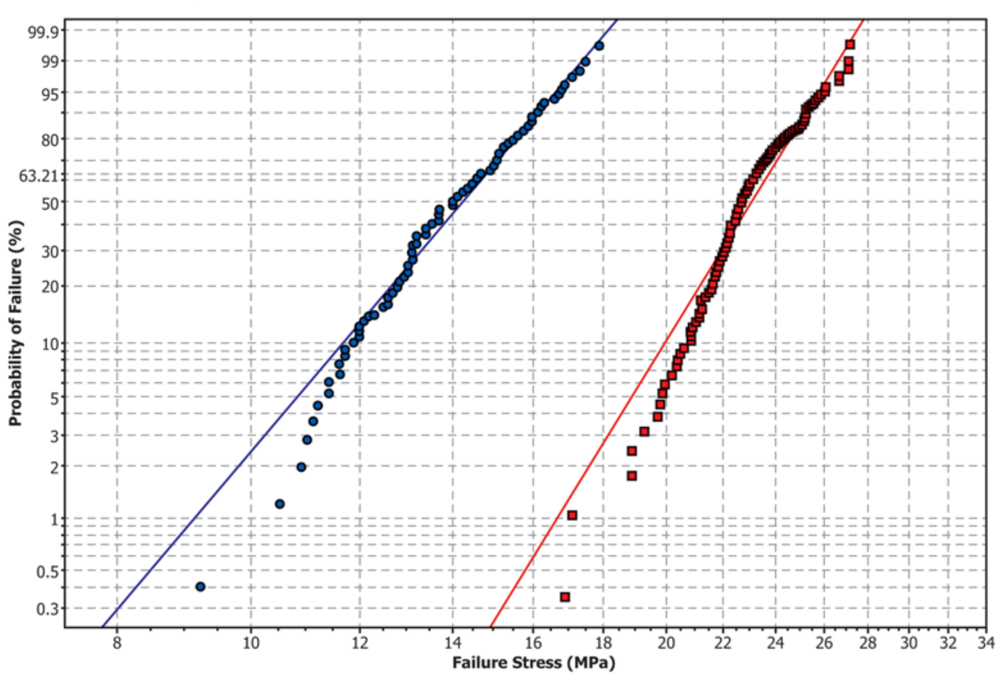

FIG. 1 Failure Strengths for Tensile Test Specimens (left) and Flexural Test Specimens (right) for a Nearly Isotropic Graphite (7)

stress in the *i*th test specimen at failure. The parameter estimates (the Weibull modulus and the characteristic strength) are determined by taking the partial derivatives of the logarithm of the likelihood function with respect to m and S_c, then equating the resulting expressions to zero. Finally, the likelihood function for the two-parameter Weibull distribution for a sample free of outlying observations is defined by the expression:

$$\mathscr{L} = \prod_{i=1}^{N} \left(\frac{\hat{m}}{\hat{S}_c}\right)\left(\frac{\sigma_i}{\hat{S}_c}\right)^{(\hat{m}-1)} \exp\left[-\left(\frac{\sigma_i}{\hat{S}_c}\right)^{\hat{m}}\right] \quad (8)$$

where r was taken equal to N in Eq 7.

7.2 The Weibull modulus (m) and the characteristic strength (S_c) are determined by taking the partial derivatives of the logarithm of the likelihood function with respect to m and S_c then equating the resulting expressions to zero. Replacing σ_i with x_i, the system of equations obtained by differentiating the log likelihood function for a censored sample is given by:

$$\hat{m} = \left[\frac{\sum_{i=1}^{r} x_i^{\hat{m}} \ln(x_i)}{\sum_{i=1}^{r} x_i^{\hat{m}}} - \frac{\sum_{i=1}^{r} \ln(x_i)}{r}\right]^{-1} \quad (9)$$

and

$$\hat{S}_c = \left[\frac{\sum_{i=1}^{r} (x_i)^{\hat{m}}}{r}\right]^{\frac{1}{\hat{m}}} \quad (10)$$

where:

r = the total number of observations (N) minus the number of outlying observations in a censored sample.

7.3 First, Eq 9 is solved first for $\hat{m}$. Obtaining a closed form solution of Eq 9 for $\hat{m}$ is not possible. This expression must be solved numerically. Subsequently, $\hat{S}_c$ is computed from Eq 10.

7.4 When a sample does not require censoring Eq 8 is used for the likelihood function. For uncensored data, the parameter estimates (the Weibull modulus m and the characteristic strength S_c) are determined by taking the partial derivatives of the logarithm of the likelihood function given by Eq 8 with respect to $\hat{m}$ and $\hat{S}_c$, then equating the resulting expressions to zero. The system of equations obtained is given by Eq 9 and Eq 10, where r = N.

7.5 An objective of this practice is the consistent statistical representation of strength data. To this end, the following procedure is the recommended graphical representation of strength data. Begin by ranking the strength data obtained from laboratory testing in ascending order, and assign to each a ranked probability of failure P_f according to the estimator:

$$P_f(x_i) = \frac{i - 0.5}{N} \quad (11)$$

where:

N = number of specimens, and
i = the *i*th datum.

Compute the natural logarithm of the *i*th failure stress, and the natural logarithm of the natural logarithm of $[1/(1 - P_f)]$ (that is, the double logarithm of the quantity in brackets), where P_f is associated with the *i*th failure stress.

7.6 Create a graph representing the data as shown in Fig. 2. Plot $\ln\{\ln[1/1(1 - P_f)]\}$ as the ordinate, and $\ln(\sigma)$ as the abscissa. A typical ordinate scale assumes values from +2 to –6. This approximately corresponds to a range in probability of failure from 0.25% to 99.9%. The ordinate axis must be labeled as probability of failure P_f, as depicted in Fig. 2. Similarly, the abscissa must be labeled as failure stress (flexural, tensile, etc.), preferably using units of MPa.

7.7 Included on the plot should be a line defined by the following mathematical expression:

$$P_f = 1 - \exp\left[-\left(\frac{x}{\hat{S}_c}\right)^{\hat{m}}\right] \quad (12)$$

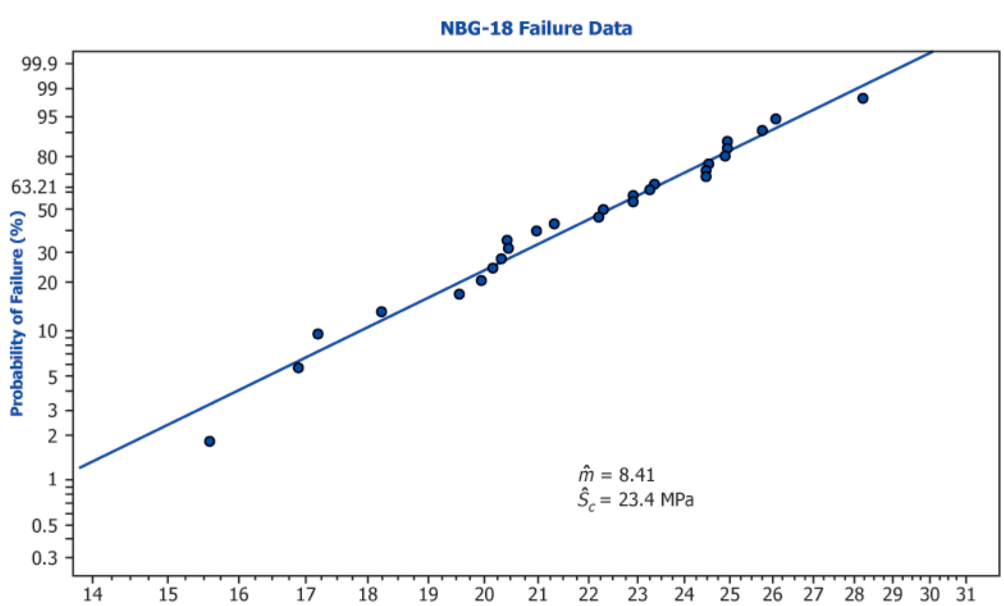

FIG. 2 Failure Strength for the Tensile Test Specimen Geometry Oriented to the Axial Direction of the Billet, End Edge Location (8)

The slope of the line, which is the estimate of the Weibull modulus *($\hat{m}$)*, and the characteristic strength ($\hat{S}_c$) should be explicitly identified, as shown in Fig. 2. The estimate of the characteristic strength corresponds to a P_f of 63.2%, or a value of zero for $\ln\{\ln[1/(1 - P_f)]\}$. A test report (that is, a data sheet) that contains information regarding the type of material characterized, the test procedure (preferably designating an appropriate standard), the number of failed specimens with the failure load and method of calculating the failure stress, the maximum likelihood estimates of the Weibull parameters, the unbiasing factor, and the information that allows the construction of 90% confidence bounds should be prepared. This data sheet should accompany the graph to provide a complete representation of the failure data. Insert a column on the graph (in any convenient location), or alternatively provide a separate table that identifies the individual strength values in ascending order. In addition, the experimentalist should include a separate sketch of the specimen geometry that includes all pertinent dimensions. An estimate of mean strength can also be depicted in the graph. The estimate of mean strength is calculated by using the arithmetic mean as the estimator:

$$\hat{\mu} = \left(\sum_{i=1}^{N} x_i\right)\left(\frac{1}{N}\right) \quad (13)$$

8. Unbiasing Factors and Confidence Bounds

8.1 Sections 8.2 through 8.4 outline methods to correct for statistical bias errors in the estimated Weibull parameters, and they outline methods to calculate confidence bounds for the distribution parameters. The procedures described herein to correct for statistical bias errors and to compute confidence bounds are appropriate only for data sets where all failures originate from an uncensored sample. The statistical bias associated with the estimate for S_c is minimal, for example, <0.3% for 20 test specimens, as opposed to ≅7 % bias for m with the same number of specimens. Therefore, this practice allows the assumption that S_c is an unbiased estimator of the true population parameter. The parameter estimate of the Weibull modulus (m) generally exhibits statistical bias. The magnitude of statistical bias depends on the number of specimens in the sample. An unbiased estimate of m shall be obtained by multiplying $\hat{m}$ by an unbiasing factor (Table 1). This procedure is discussed in the following sections. Statistical bias associated with the maximum likelihood estimators presented in this practice can be reduced by increasing the sample size.

8.2 An unbiased estimator produces nearly zero statistical bias between the value of the true parameter and the point estimate. The amount of deviation can be quantified either as a percent difference or with unbiasing factors. In keeping with the accepted practice in the open literature, this standard quantifies statistical bias through the use of unbiasing factors, denoted here as *UF*. Depending on the number of specimens in a given sample, the point estimate of the Weibull modulus ($\hat{m}$) may exhibit significant statistical bias. An unbiased estimate of the Weibull modulus (denoted as $\hat{m}_u$) is obtained by multiplying the biased estimate with an appropriate unbiasing factor. Unbiasing factors for $\hat{m}$ are listed in Table 1. The example in section 9.2 demonstrates the use of Table 1 in correcting a biased estimate of the Weibull modulus. As a final note, this procedure is not appropriate for censored samples. The theoretical approach was developed for uncensored samples.

TABLE 1 Unbiasing Factors for the Maximum Likelihood Estimate of the Weibull Modulus (9)

Number of Specimens, *N*	Unbiasing Factor, *UF*	Number of Specimens, *N*	Unbiasing Factor, *UF*
5	0.700	42	0.968
6	0.752	44	0.970
7	0.792	46	0.971
8	0.820	48	0.972
9	0.842	50	0.973
10	0.859	52	0.974
11	0.872	54	0.975
12	0.883	56	0.976
13	0.893	58	0.977
14	0.901	60	0.978
15	0.908	62	0.979
16	0.914	64	0.980
18	0.923	66	0.980
20	0.931	68	0.981
22	0.938	70	0.981
24	0.943	72	0.982
26	0.947	74	0.982
28	0.951	76	0.983
30	0.955	78	0.983
32	0.958	80	0.984
34	0.960	85	0.985
36	0.962	90	0.986
38	0.964	100	0.987
40	0.966	120	0.990

8.3 Confidence intervals represent uncertainty about a point estimate of a population parameter. Confidence interval widths of both Weibull parameters decrease with more specimens. The confidence bounds are the associated percentiles of the distribution of parameter pivotal quantities obtained via Monte Carlo simulation. For example, the 90 % confidence bound on the Weibull modulus is obtained from the 5 and 95 percentiles of the distribution of the ratio of the pivotal quantity, $\frac{\hat{m}}{m}$. Thoman (1969) noted the pivotal function, $\frac{\hat{m}}{m} = \hat{m}_u$, which holds regardless of the true modulus. Where q^* represents the *th percentile of the distribution of $\frac{\hat{m}}{m}$ for the appropriate sample size, the confidence intervals for the true modulus are obtained as follows:

$$q_{LB} < \hat{m}_U < q_{UB} \quad (14)$$

$$q_{LB} < \frac{\hat{m}}{m} < q_{UB} \quad (15)$$

$$\frac{\hat{m}}{q_{UB}} < m < \frac{\hat{m}}{q_{LB}} \quad (16)$$

Thus, to get the confidence bounds on the m, divide the biased estimate by q^* coefficients corresponding to the appropriate percentile and sample size. Table 2 provides the normalizing coefficients. The example in 9.2 demonstrates the use of Table 2 in constructing the upper and lower bounds on m. Note the biased estimate of the Weibull modulus must be used here.

8.3.1 Table 2 has values for up to 120 specimens. Equations valid for up to 500 were numerically fitted to the data **(10)**. The equations for the bounds are given by: