

ON THE SUITABILITY OF VARIOUS STATISTICAL MODELS FOR PREDICTION OF GRAPHITE FAILURE STRENGTH

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ABSTRACT

Weibull model was employed to predict size-effect in tensile specimens and flexural strength. Weibull parameters estimated from the test data of small-sized tensile specimens were utilized for prediction of tensile and flexural strength of large sized specimens. Numerical predictions were then compared with experimental data. It was observed that this model over-predicts size effect in tension. However, incorporation of grain size effect in Weibull model, allows a more realistic simulation of size effects. In addition, Rose and Tucker model and Burchell model which account for microstructural parameters such as particle size, porosity, fracture toughness etc. were used for predicting tensile and flexural strength. The suitability of the above-mentioned models was studied by comparing the numerically predicted failure strength with the experimental data available in the literature. Rose and Tucker model predicts the mean failure strength accurately, but fails to predict the entire strength distribution. Burchell model also predicts mean failure strength accurately but does not give accurate predictions at the lower tail of the strength distribution. Apart from analyzing strength data available in the literature, a large number of graphite specimens was tested at room temperature under tensile, four point bending and three point bending load. Weibull parameters were estimated from the strength data of large sized tensile specimens. By utilizing these parameters, four point and three point bend strength distributions were predicted and subsequently, they were compared with the experimentally observed strength distributions. Weibull model was observed to over-predict the flexural strength.

INTRODUCTION

High density and high purity grade of graphite is proposed to be used as a structural material and reflector in Compact High Temperature Reactor (CHTR), India. Inside the reactor core the graphite components would be subjected to combined loadings resulting from thermal loads, mechanical loads and the stresses due to irradiation induced dimensional changes. In addition, seismic and dead loads would also be significant for many structural components. These stresses, acting either alone or in combination should not be allowed to cause structural failures. It is well recognized that the macroscopic fracture process in graphite is intimately related to the microstructural features which influence the process of crack initiation and propagation. Due to the random nature of several microstructural features, the observed failure strength of graphite is statistical in nature. In this regard, detailed numerical as well as experimental studies were conducted on various probabilistic models viz. Weibull, Rose & Tucker and Burchell model that have been proposed in the literature to address strength related issues in graphite. Weibull model uses the experimental data of failure loads of small size specimens to predict the fracture load of actual components. This model does not require any microstructural details and, thus, is simple to use. Rose & Tucker and Burchell models, on the other hand, do account for the microstructural details. As a result, these models are expected to simulate the actual failure mechanism in graphite in a more realistic manner. Rose & Tucker model considers crack initiation from the filler particles when the particle cleavage stress is exceeded. This model does not account for stress intensification due to pre-existing flaws. However, Burchell model incorporates fracture mechanics criteria and considers crack initiation

from pre-existing flaws. This paper includes the studies on these statistical models to assess their suitability to predict the failure strength of graphite accurately.

NUMERICAL PREDICTION OF GRAPHITE FAILURE STRENGTH

Prediction Of Tensile And Bending Strength Using Weibull Model

In order to predict failure strength of graphite, Weibull model[6] was employed. Instead of accounting for the microstructural details explicitly, it utilizes experimental data for evaluation of Weibull parameters. This model was used to predict the effect of type of loading and the so-called size effect on strength of graphite. To assess the performance of this model, experimental data taken from Price's report[4] on extruded near-isotropic petroleum coke based nuclear graphite (Great Lakes Carbon Corporation grade H-451) was analyzed. Weibull parameters (m and σ_0) were estimated from tensile strength data of small sized specimens using maximum likelihood method[1]. Weibull's two parameter distribution function for failure probability is described as follows.

$$P_f = 1 - e^{-\int \left(\frac{\sigma}{\sigma_0}\right)^m dV} \quad (1)$$

Where m is Weibull modulus and σ_0 is scale parameter. When a specimen is subjected to bending load, stresses generated are not uniform throughout, but vary along the length of the specimen as well as in the direction of applied load. In this case, to calculate probability of failure of the specimen, integral as shown in Equation 1 needs to be evaluated over the entire volume where tensile stress is present. Equation 1 can be rewritten as:

$$P_f = 1 - e^{-\left(\frac{\sigma_f}{\sigma_0}\right)^m V_e} \quad (2)$$

Where σ_f is maximum fiber stress in tension and V_e is effective volume given as:

$$V_e = \int \left(\frac{\sigma}{\sigma_f}\right)^m dV \quad (3)$$

For predicting four point bend strength, effective volume was calculated with the help of Equation 3 and predictions were made using Equation 2. Figure 1 shows the effect of type loading on the predicted failure strength. The specimens loaded in four point bending failed at higher stress as compared to that of tensile specimens. This behaviour is ascribed to lesser volume of bend specimens subjected to maximum tensile stress. This model predicts higher strength in flexure but fails to predict the entire strength distribution. The effect of specimen size on failure strength under tensile load is shown in Figure 2. As expected, large tensile specimens failed at lesser stress than the small ones. It is evident from Figure 2 that the size effect is over-predicted. The difference between the predicted and experimental strength for 50% failure probability is more than 20%. Weibull model[6] predicts continuously increasing strength as the volume of specimen decreases. But, when the least dimension of the specimen is of the order of grain size, sudden decrease in the strength has been observed experimentally. To account for this effect, Weibull model was modified by Ho[3] as given by Equation 4.

$$P_f = 1 - e^{-\int \left(\frac{\sigma}{\sigma_0 f_1 f_2}\right)^m dV} \quad (4)$$

Where f_1 and f_2 are parameters which depend on specimen dimensions and grain size.

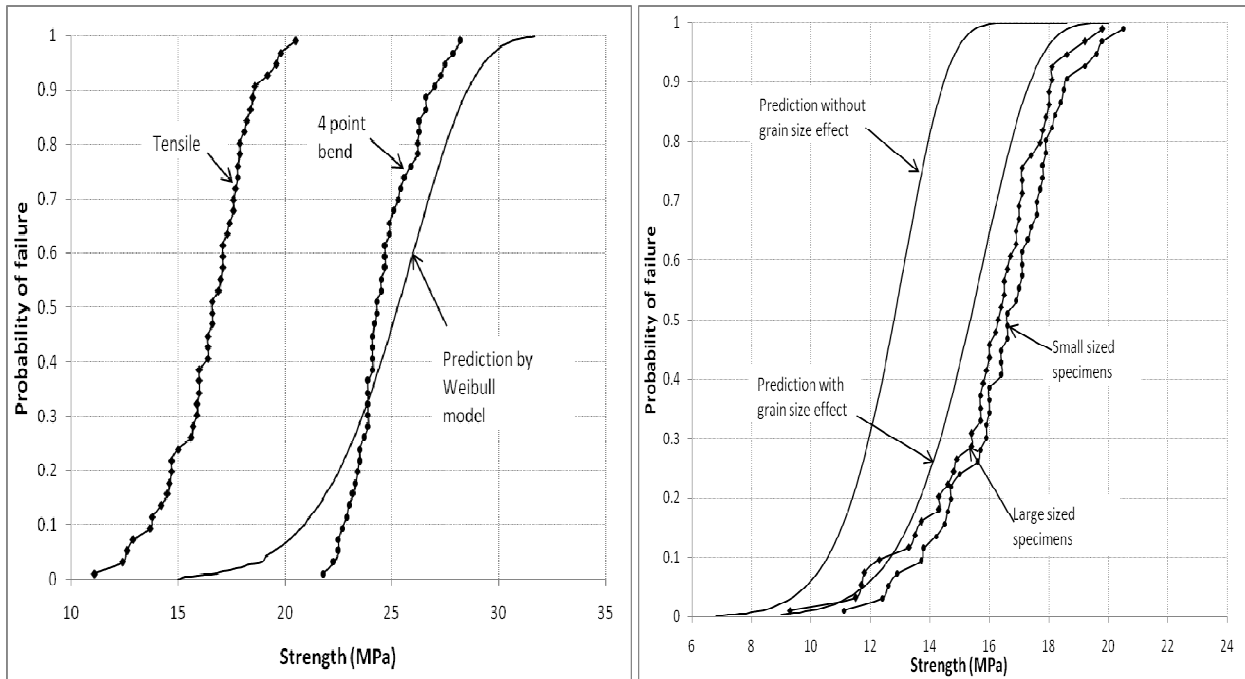


Figure 1. Effect of type of loading on failure strength. Figure 2. Prediction of size effect in tension.

This modified Weibull model was then employed to predict size effect. As can be seen in Figure 2, incorporation of grain size effect on failure strength improves the prediction of size effect and the difference reduces to less than 10% as shown in Table 1. The effect of grain size on the predicted four point bend strength could not be studied because test data on bend specimens of different sizes was not available in Price's report[4].

Table 1: Comparison of predicted and experimental strength data for 50% failure probability.

Type of specimen	Dimensions (mm)	Experimental Strength* (MPa)	Predicted Strength Without Grain Size Effect (MPa)	Predicted Strength With Grain Size Effect (MPa)	% Difference (Without grain size effect)	% Difference (With grain size effect)
Large Sized Tensile	d=12.4, l=76	16.3	12.8	15.4	-21.47	-5.52
Four Point Bend	d=6.4, l=51	24.3	25.1	-	3.29	-

Notes: *The experimental strength values quoted here are the values for 50% failure probability published by R. J. Price (1976).

In this table, the strength was predicted based on experimental data published by R. J. Price (1976)

Effect of microstructural parameters on graphite failure strength

It is well recognized that the failure strength of graphite depends on various microstructural parameters. In view of this, Rose and Tucker model[5] which accounts for some of the microstructural parameters was considered. This model assumes that crack initiation takes place due to cleavage of filler particle. When normal component of applied tensile stress on critical planes (basal planes) exceeds the critical cleavage stress, the particle is said to have cleaved. Cracks are assumed to develop on planes normal to the axis of principal stress. The plane will fail when a large particle cleaves and sufficient numbers of adjacent binder particles also cleave to form a critical crack according to the criterion of LEFM. Pores are treated in the model as particles with zero cleavage strength. The probability of failure of the component is then the probability that any one of the planes will contain one such defect.

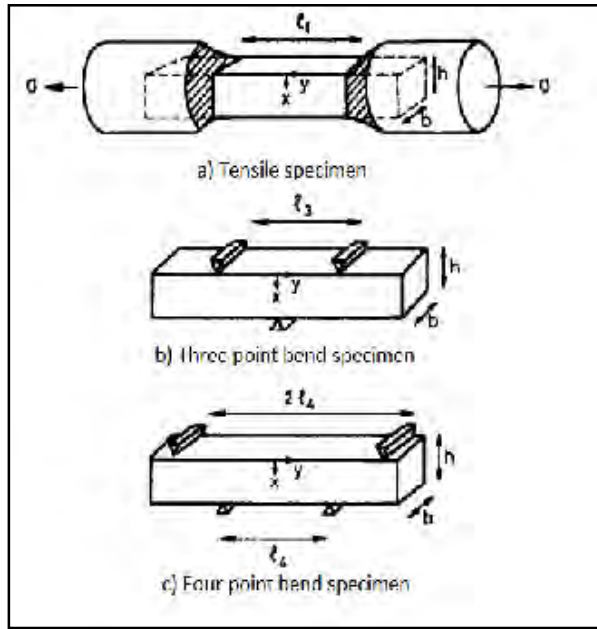


Figure 3. Type of specimens[5] analyzed in present work.

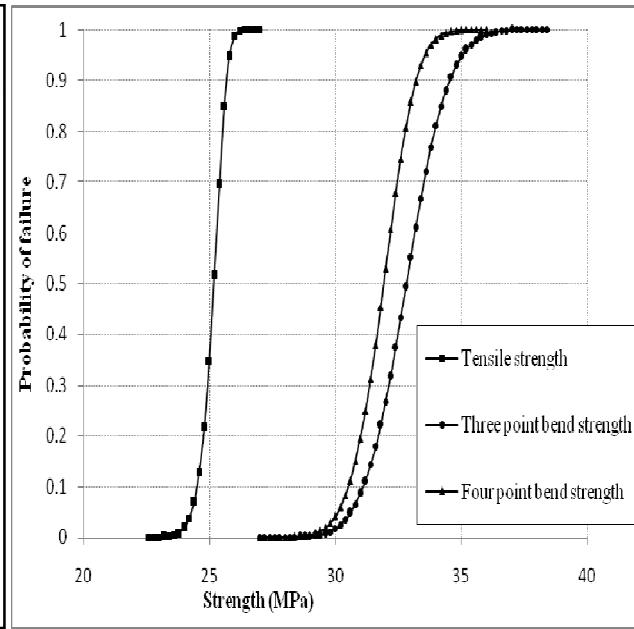


Figure 4. Prediction of tensile, three-point bend and four point bend strength using Rose & Tucker model[5].

This model was employed to predict strength of tensile, three-point bend and four-point bend specimens shown in Figure 3. Dimensions of these specimens are provided in Table 2. Input parameters to this model such as particle size, particle cleavage stress, number of particles per unit volume, critical stress intensity factor (K_{Ic}) and fractional porosity available in the literature[5] were utilized. Figure 4 shows predicted failure probability vs. strength curves for tensile, three point and four point bend loadings. As apparent from Figure 4, predicted tensile strength is lower than flexural strength whereas four point bend strength lies in between tensile and three point bend strengths. This variation in strengths can be attributed to volume dependence of strength of graphite. Since volume of three point bend specimens subjected to higher tensile stress is very small, probability of finding any weak flaw in this volume is less and hence three point bend specimens fail at higher loads as compared to tensile specimens. The scatter in the strength also depends on volume under tensile stress. As can be seen in Figure 4, probability of failure vs. strength curve for tensile strength is steeper as compared to that for flexural strength and this implies less scatter in tensile strength. Predicted mean strengths were compared with the experimentally observed mean strengths and the results are provided in Table 2.

Table 2: Comparison of predicted mean strength by Rose & Tucker model[5] with experimental data.

Type of Loading	Specimen Dimensions (mm)	Predicted Strength (MPa)	Experimental Strength* (MPa)	No. of Tests	%Difference
Tension	$l_1=30, b=10, h=10$	25.13 ± 0.5	20.6 ± 1.8	7	22
3 Point Bend	$l_3=19, b=8, h=8$	32.83 ± 1.4	34.09 ± 0.9	100	-3.7
4 Point Bend	$l_4=19, b=8, h=8$	31.89 ± 1.1	30.1 ± 1	12	5.95

Note: *The experimental strength values quoted here are the mean values published by Rose & Tucker (1982).

Rose and Tucker model[5] neither accounts for stress intensification due to pre-existing flaws, nor takes into account the pore size distribution. However, Burchell model[2] does address these issues. Tensile strengths for three different grades of graphite viz. H-451, IG-110 and AXF-5Q were predicted employing Burchell model[2] and subsequently compared with the experimentally observed data available in the literature. Input parameters to this model such as mean particle size, mean pore size, standard deviation of pore size distribution, particle fracture toughness, bulk fracture toughness, number of pores per unit volume, volume of specimen under loading etc. available in the literature[2] were utilized. Figure 5 shows the predicted and experimentally observed tensile strength distribution curves for the above mentioned grades of graphite.

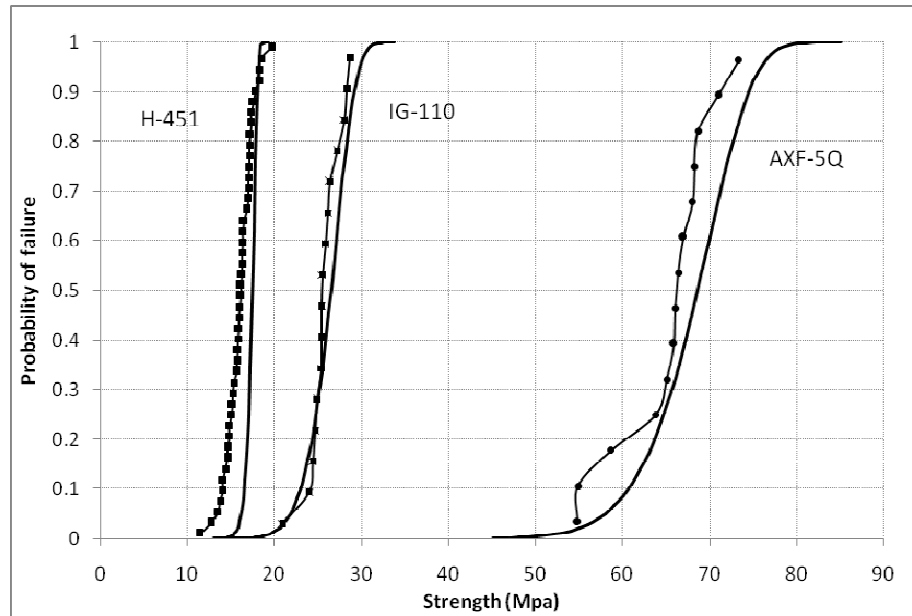


Figure 5. Comparison of predicted and experimentally observed strengths using Burchell model[2].

Table 3: Comparison of predicted tensile strength using Burchell model[2] with experimental data for 50% failure probability.

Grade of Graphite	Experimental Tensile Strength* (MPa)	Predicted Tensile Strength (MPa)	% Difference
H 451	16.12	17.45	8.25
IG-110	25.45	26.5	4.13
AXF-5Q	66.19	68.6	3.64

Note: *The experimental strength values quoted here are the strength values for 50% failure probability published by T. D. Burchell (1996).

Figure 5 shows that predicted strength distributions are reasonably accurate and the difference between mean values of predicted and experimentally observed strengths is within 10%. These reasonably accurate predictions made by Burchell model[2] can be ascribed to its sound physical basis. However, this model does not give accurate predictions at the lower tail of the strength distribution.

MECHANICAL TESTING OF GRAPHITE SPECIMENS

To assess the applicability of statistical strength prediction models such as Weibull model[6], a large number of tests was carried out on a high purity and high density graphite. In this work, around 25 large sized tensile specimens (D = 25.4 mm) fabricated as per ASTM C-749 and 30 flexural specimens fabricated as per ASTM C-651 were tested on a computer controlled servo-hydraulic testing machine having a load capacity of 20 kN. Out of 30 flexural specimens, 15 specimens (25 X 25 X 250 mm) were subjected to four point bending whereas remaining 15 (25 X 50 X 250 mm) were subjected to three point bending load. Figure 6 shows the typical tensile test set up.



Figure 6. Experimental setup for tensile test.

Test results

The summary of test data of tensile and flexural specimens is given in Table 4. The flexural strengths were calculated as per ASTM C-651. The specimens subjected to three point bending load failed at higher stress as compared to that of four point bend specimens. In addition to this, strengths of both three point as well as four point bend specimens were higher than the strength of large sized tensile specimens. This behaviour also known as ‘loading effect’ is ascribed to non-uniform stress distribution in flexural specimens. Due to non-uniform stress distribution, the effective volume of flexural specimens subjected to maximum tensile stress is less and hence flexural specimens fail at higher stress.

Table 4: Summary of tensile and flexural strength data based on tests by the authors.

Type of Test	No. of Tests	Mean Strength (MPa)	Std. Deviation (MPa)	Coefficient of Variation (%)
Tensile	25	25.4	1.8	7.08
Four Point Bend	15	26.36	3.41	12.93
Three Point Bend	15	29.87	1.49	4.99

ANALYSIS OF EXPERIMENTAL FAILURE STRENGTH OF GRAPHITE USING WEIBULL MODEL

Estimation of Weibull parameters

The strength data of large sized tensile specimens was utilized for estimating Weibull parameters i.e. Weibull modulus (m) and scale parameter (σ_0). These parameters were estimated by maximum likelihood method as recommended by ASTM C-1239. These parameters are shown below.

Estimated Weibull modulus (\tilde{m}) = 15.74
 Estimated characteristic strength ($\tilde{\sigma}_0$) = 26.21 MPa

Figure 7 shows the experimentally observed tensile strength of large sized specimens and Weibull's fit to this data.

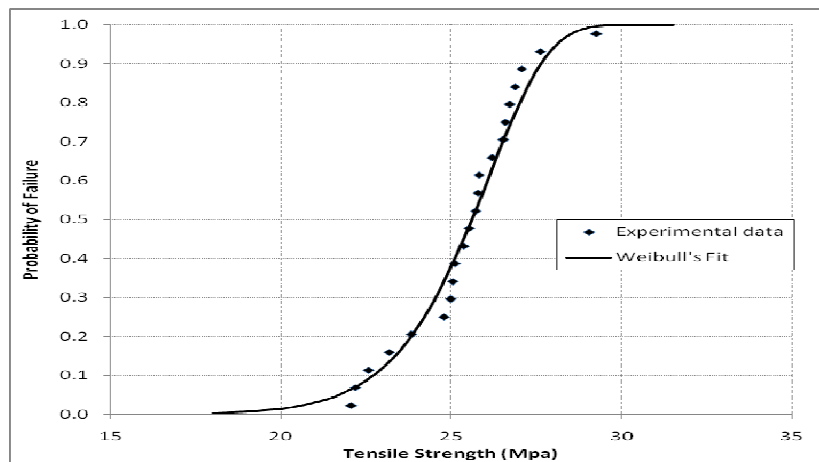


Figure 7. Experimental tensile strength distribution and Weibull's fit.

The characteristic strength (σ_0) is dependent on specimen geometry and volume. Therefore, to predict failure strength of specimens with different geometry and size, scale parameter (σ_0) which is independent of specimen geometry and size was calculated using Equation 5.

$$\sigma_0 = \sigma_\theta V_e^{\frac{1}{m}} \quad (5)$$

Scale parameter ($\tilde{\sigma}_0$) = 49.86 MPa.(mm³)^{1/15.74}

Prediction of four point bend strength and comparison with experimental data

The stress distribution in a beam subjected to four point bending load is non-uniform. Between inner supports, stress varies in the direction of applied bending load and it is constant along the length of the specimen but in the outer span, it varies in both the directions. Hence equivalent volume subjected to uniform stress equal to maximum outer fiber stress which will have same risk of rupture as that of four point bend specimen can be calculated from Equation 3. For four point bend specimen having rectangular cross-section, effective volume is given as

$$V_e = \frac{WTL_o}{4(m+1)} \left[1 + \frac{1}{(m+1)} \right] \quad (6)$$

Where ‘W’ is width, ‘T’ is thickness, ‘L_o’ is outer span for four point bend specimen and ‘m’ is the estimated Weibull modulus.

Weibull parameters estimated in the earlier section from tensile strength data were used for predicting four point bend strength. Effective volume was calculated from Equation 6 and subsequently, four point bend strength was predicted using Equation 2. Figure 8 shows the experimentally observed four point bend strength as well as Weibull’s prediction.

It is evident from Figure 8 that Weibull model[6] over-predicts entire four point bend strength distribution. Experimentally observed mean four point bend strength is 26.36 MPa whereas the mean strength predicted by Weibull model[6] is 29.77 MPa.

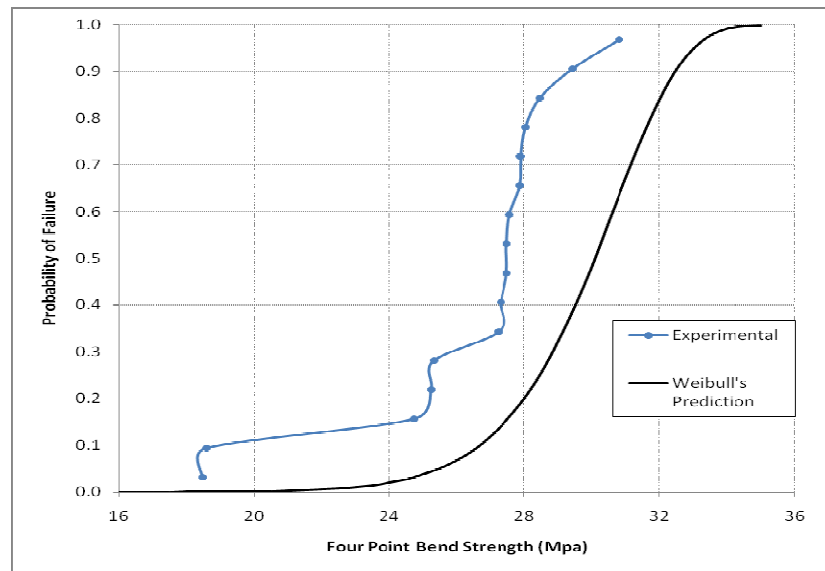


Figure 8. Experimental and predicted four point bend strength distribution.

Prediction of Three Point Bend Strength and Comparison with Experimental Data

The stress distribution in a beam subjected to three point bending load is non-uniform and varies along the length of the specimen as well as in the direction of applied load. Therefore, equivalent volume subjected to uniform stress equal to maximum outer fiber stress can be calculated from Equation 3. For three point bend specimen having rectangular cross-section, effective volume is given as

$$V_e = \frac{WTL_o}{2(m+1)^2} \quad (7)$$

Where ‘W’ is width, ‘T’ is thickness, ‘L’ is total span for three point bend specimen and ‘m’ is the estimated Weibull modulus.

Weibull parameters estimated in the earlier section from tensile strength data were used for predicting three point bend strength. Effective volume was calculated from Equation 7 and subsequently, three point bend strength was predicted using Equation 2. Figure 9 shows the experimentally observed three point bend strength as well as Weibull’s prediction. The mean three point bend strength predicted by Weibull model is 32.72 MPa whereas experimentally observed strength is 29.87 MPa. Similar to four point bend strength, Weibull model[6] over-predicts entire three point bend strength distribution too.

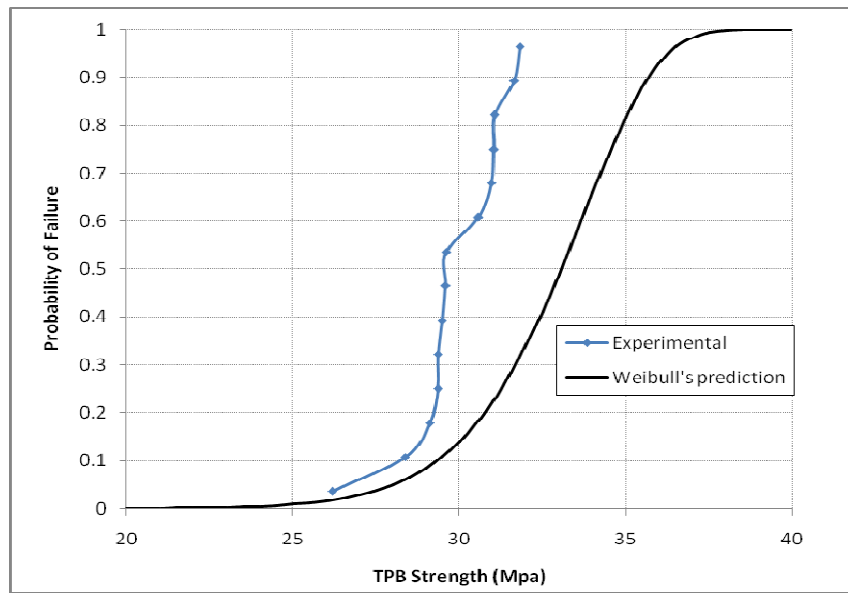


Figure 9. Experimental and predicted three point bend strength distribution.

Table 5: Comparison of experimental and predicted flexural strength by Weibull model for 50% failure probability.

Type of Loading	Experimental Strength (MPa)	Predicted Strength (MPa)	% Difference
Four Point Bend	27.46	30.07	9.5
Three Point Bend	29.6	33.06	11.69

CONCLUSION

Initial part of this paper contains the analysis of graphite failure strength data available in the literature by Weibull, Rose & Tucker and Burchell models whereas the later part contains the analysis of test data generated by the authors using Weibull model.

Graphite is a quasi-brittle material and its failure strength shows scatter. Therefore, the strength prediction model should account for this statistical nature. Various statistical models considered in this paper differ in the way they account for the mechanism of failure in graphite. Analysis of graphite failure strength data available in the literature shows that Weibull model predicts mean failure strength of 4-point bend specimen with reasonable accuracy, but fails to predict entire strength distribution accurately. For tensile specimens, it under-predicts the strength of large sized specimens if grain size correction is not considered. Although incorporation of grain size effect improves predicted failure strength, entire distribution of strength can not be predicted accurately. Rose and Tucker model and Burchell model predict uni-axial strength accurately. However, these models fail to predict lower tail of the strength distribution accurately.

Graphite is not perfectly brittle and shows rising R-curve behaviour. Weibull model is expected to give conservative results as this model considers that only crack initiation is sufficient to cause ultimate failure and it does not account for rising R-curve behaviour. Analysis of graphite failure strength data generated in-house however shows that Weibull model gives unconservative predictions of four point and three point bend strengths. Further studies are required to confirm these new findings and the applicability of Weibull model needs to be looked upon for numerical predictions of graphite flexural strength using the Weibull parameters evaluated from tensile test data.

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