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A Note on Statistical Strength of Carbon Nanotubes

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Abstract: This note aims to relate the measured strength statistics of individual carbon nanotubes (CNTs) to the physics of brittle fracture and the weakest link model. By approximating an arbitrary flaw size distribution with a segmented power law, an effort is made to extend applicability of the Weibull distribution to arbitrary flaw populations, which explains why the Weibull distribution fits the experimental data of CNTs and many other brittle materials, and why in other cases it is not so clear. A generalized Weibull distribution is proposed to account for all non-asymptotic cases. The published CNT testing data are analyzed, and finally a major issue present in existing interpretation of CNT bundle testing data is clarified.

Keywords: Strength, Power law, Weibull distribution, Carbon nanotubes.

1 Introduction

Characterization and modeling are two fundamental scientific approaches. Characterization of scattering or statistics of fracture strength must be empirical, on the one hand. But this approach alone certainly does not satisfy one who distinguishes probabilistic modeling from statistical analysis, especially in dealing with medium-to-high consequence events where the sample size is small. In his 1968 article Freudenthal noted clearly "the inherent weakness of this procedure (i.e. statistical analysis) is the impossibility of discriminating between different statistical distribution functions on the basis of the moderate number of test replications usually available" [Freudenthal (1968)]. Perhaps addressing some debates constantly occurring at that time, he warned that "Thus, for instance, to distinguish between

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a logarithmic normal and an extremal distribution with a reasonable degree of confidence that would justify extrapolation, several thousand test results would have to be available" [Freudenthal (1968)]. Not by coincidence, there were similar debates and discussions [Lu, Danzer, Fischer (2002); Pugno and Ruoff (2006); Klein (2007); Barber, Andrews, Schadler, Wagner (2005); Lu (2005); Wagner, Barber, Andrews, Schadler (2008)] arising recently on statistical strength of carbon nanotubes (CNTs), in which case actually only dozens of CNT tensile testing specimens were available.

Therefore, on the other hand, probabilistic modeling of scattering or statistics of fracture strength is required. Freudenthal suggested that "An alternative approach is to establish physically relevant probability models, which are then used for extrapolation… not on the basis that the distributions they produced can be fitted to existing test results (which is not a sufficient condition), but that they are germane to the phenomenon" (Freudenthal, 1968).

In Section 2 of this note, an effort is made in attempt to relate the measured strength statistics of individual CNTs to the physics of fracture and the weakest link model, and explain why Weibull distribution fits the experimental data of CNTs and many other brittle materials, and why in other cases it is not so. In section 3, published CNT testing data are analyzed and commented. A major issue present in existing interpretation of CNT bundle testing data is clarified.

2 Power Law and Weibull Distribution

2.1 Asymptotic Weibull distribution

The flaw size c in a CNT is characterized as half of the flaw dimension along the cross section of the CNT, which has a lower bound a the lattice space, and an upper limit c_{max} the half length of the CNT perimeter. A weakest link element of a CNT is defined as the one with the strength statistically independent from any other elements especially the upper and lower adjacent neighbors. The minimum length of the element ℓ varies depending on the largest flaw considered. In the asymptotic analysis the lower tail of the strength distribution of the element corresponds to the flaw size approaching c_{max}. The minimum length ℓ then is $\sim \pi D$ with D the diameter of the CNT.

Denote $P_\ell^c(c)$ the probability of a flaw appearing in the weakest link element with the size less than or equal to c. For an arbitrary distribution function $P_\ell^c(c)$, we assume, as is typical, that its large flaw tail can be approximated with a power law, i.e.

$$1 - P_\ell^c(c) \approx A(c_{\max} - c)^\alpha, \text{ when } c \to c_{\max} \tag{1}$$

where $\alpha > 0$ is the slope near the c_{max} (Fig.1), and A is a constant. Note the distribution $P_\ell^c(c)$ depends on ℓ the length of the weakest link element that is chosen. The form in Eq. (1) is not universal and other forms can arise, for instance, when initial flaws are non-contiguous, containing bridging bonds, as seen in 1D and 2D networks [Li and Duxbury (1987); Phoenix and Beyerlein (2000)]. When the concentration of large flaws is extremely low, i.e. negligible effect of flaw interaction, by generalizing Griffith's criterion the tensile strength of a CNT is formulated as

$$\sigma = \frac{T}{\psi\, c^{1/\beta}} \tag{2}$$

where T serves as the toughness, β is the exponent on the flaw size, equal to 2 for cracks much longer than the individual bonds, and ψ the factor to account for the finite size and curvature. In the extreme case when the flaw size approaches c_{max}, the effect of ψ dominates. Following the familiar strength formula for a finite width plate containing a crack, the CNT strength is approximated as

$$\sigma \propto \sqrt{\frac{c_{\max} - c}{c_{\max} c}}\text{, when } c \to c_{\max} \tag{3}$$

which by using Taylor's series can be expanded as

$$\sigma \propto \frac{\sqrt{c_{\max} - c}}{c_{\max}} + O\left(\left(\sqrt{\frac{c_{\max} - c}{c_{\max}}}\right)^3\right), \text{ when } c \to c_{\max} \tag{4}$$

With (1) and (4), the lower tail of the strength distribution for a weakest link element is obtained as $P_\ell(\sigma) \propto \sigma^{2\alpha}$, when $\sigma \to 0$, or explicitly written as $P_\ell(\sigma) = \left(\frac{\sigma}{\sigma_0}\right)^{2\alpha}$ with σ_0 the scale parameter. According to the theory of extreme value statistics [Fisher and Tippett (1928); Gumbel (1958)] when the total number of element in a CNT N_e becomes sufficiently large, the strength distribution of the CNT converges to the Weibull distribution asymptotically

$$P_W(\sigma) = 1 - \exp\left(-N_e \left({}^{\sigma}/_{\sigma_0}\right)^{2\alpha}\right) \tag{5}$$

While the derivation of the Weibull distribution (5) is mathematically rigorous and consistent with the physics of failure by the weakest link model, it in this case has little value of application since either the strength is so low or the event is so rare that such an asymptotic result itself is generally out of engineering interest. Our interests lie in the strength distribution for a finite length CNT, and how good the form (5) is to represent it. In other words, if we are going to use (5), how fast the true distribution converges to the Weibull distribution (5) as CNT length increases.

Below we will discuss such a non-asymptotic Weibull distribution. Relevant studies on the size effect of the weakest link model can be found in [Wagner (1989); Phoenix SL, Ibnabdeljalil M, and Hui (1997); Mahesh, S, Beyerlein, IJ, Phoenix (1999); Yu, Lourie, Dyer, Moloni, Kelly, Ruoff (2000); Bhattacharya B and Lu (2006); Bažant ZP and Pang (2007)].

2.2 Non-asymptotic Weibull distribution

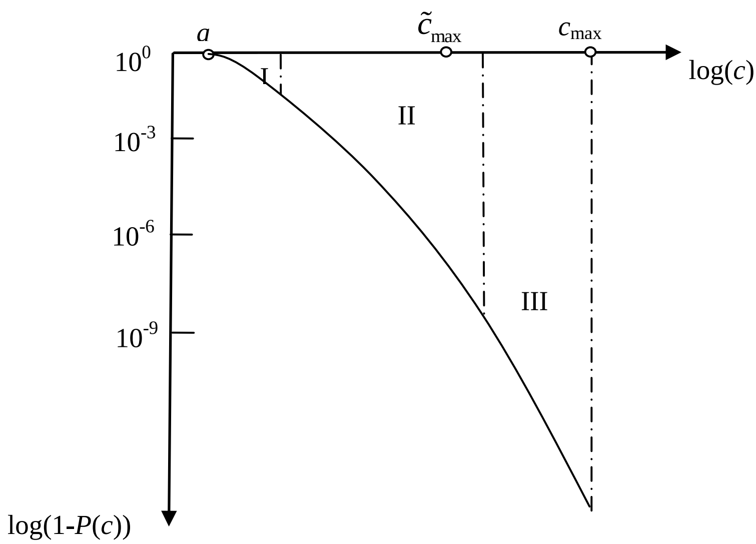

Figure 1: Schematic of a log-log plot of the flaw size distribution in a CNT.

An important point presented above in the asymptotic case is that the Weibull distribution can be related to the power law approximation of flaw size statistics near the tail. In fact such an approximation can be extended to the whole domain of $P(c)$, the log-log slope of which varies slowly, by using a number of power law segments, e.g. one segment to approximate Region II in Fig.1. As the number of test specimens in engineering is typically hundreds or even dozens (e.g. in the CNT case), the power law segments reached by a test is normally located in Region II (Fig.1), which is usually characterized by one or two power law segments. Note that since $P(c)$ is monotonic all the exponents of the segments in the log-log plot are non-negative. Region I and III in Fig. 1 correspond to extreme high and extreme low strength tails, respectively, which are usually not assessable in actual engineering experiments. In other words, the sampled largest flaw size terminates at $\tilde{c}_{\max}$ much smaller than $c_{max.}$, and the distribution at this sampled tail can always be approximated as

$$1 - P_\ell^c(c) \approx Ac^{-\alpha} \tag{6}$$

when c is in the vicinity of $\tilde{c}_{\max}$, where $A > 0$ and $\alpha > 0$. Note while Eq. (6) can approximate any distribution at vicinity of $\tilde{c}_{\max}$, the power law approximation becomes useful only when the exponent α is close to a constant over a statistically sizable segment. With (2) and (6), the sampled lower tail of the strength distribution for a weakest link element is obtained as

$$P_\ell(\sigma) = \left(\sigma/\sigma_0\right)^{\alpha\beta} \tag{7}$$

where $\sigma$is the strength corresponding to the flaw size at vicinity of $\tilde{c}_{\max}$. The scale parameter

$$\sigma_0 = \frac{T}{\psi A^{1/\alpha\beta}} \tag{8}$$

varies with the length of the element. Now with the power law strength distribution given, let us see how good the Weibull distribution (5) is to approximate the true weakest link distribution for a finite N_e

$$P_L(\sigma) = 1 - \left[1 - \left(\sigma/\sigma_0\right)^{\alpha\beta}\right]^{N_e} \tag{9}$$

When the length of the weakest link element is taken as the minimum length, the scale parameter σ_0 is valued at the three-digit level, e.g. the scale parameter of a weakest link element with the minimum length 50 nm and diameter D = 20 nm is found to be 213 GPa (see Section 3.1). According to the CNT tensile testing data [Klein (2007); Yu, Lourie, Dyer, Moloni, Kelly, Ruoff (2000)], the Weibull modulus m=$\alpha\beta$ is between 2$\sim$3. By letting $\sigma_0 = 150$GPa, the absolute relative error of the approximation $\frac{|P_W(\sigma)-P_L(\sigma)|}{P_L(\sigma)}$ is plotted in Fig.2 (see Appendix). The figure shows with 50 weakest link elements, the Weibull approximation is good enough with the maximum relative error about 0.6%.

In the above exposition the sampled tail is supposed to be located in Region II (Fig. 1), i.e. change of the slope within the region is slow and one modulus is statistically good enough, which is the case in CNTs according to the reported data [Barber, Andrews, Schadler, Wagner (2005); Klein (2007); Yu, Lourie, Dyer, Moloni, Kelly, Ruoff (2000)]. However, with increase of the number of testing specimens, the sampled tail will eventually invade into Region I or III. If the slopes of these regions are obviously different, then a bi-modal or multi-modal Weibull

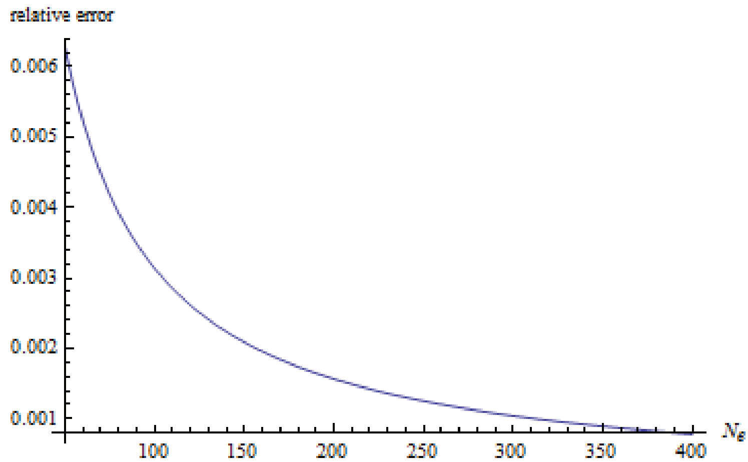

Figure 2: Relative errors of the Weibull distribution (5) when approximating the weakest link distribution (8)

distribution will become necessary, which has two or more moduli corresponding to several strength regions.

Based on the above exposition, we propose the following generalized Weibull distribution to cover all the non-asymptotic cases, as

$$P_W(\sigma) = 1 - \exp\left(-N_e \left({}^{\sigma}/_{\sigma_0}\right)^{m(\sigma)}\right) \tag{10}$$

where the Weibull modulus m is generally a function of strength σ. For an intermediate value of the strength σ corresponding to the maximum defect located in Region II, $m$is treated approximately as a constant, i.e.

$$m_{II} = \alpha_{II}\beta_{II} \tag{11}$$

where β_{II} is 2 that fracture mechanics applies approximately, and α_{II} is a constant between approximately 1∼ 1.5 based on CNT tensile testing data [Klein (2007); Yu, Lourie, Dyer, Moloni, Kelly, Ruoff (2000)]. When the strength σ in (10) increases entering into the high strength region, the maximum defect size moves into Region I, and as shown in Fig. 1 the Weibull modulus α_I continuously decreases being not a constant. For the upper strength limit at the left tail in Region I, the modulus can be expressed as

$$m_I^+ = \alpha_I^+\beta_I^+ \tag{12}$$

with superscript + denoting the upper strength limit. In this single lattice defect case, m_I^+ is expected to be smaller than m_{II}, indicating greater statistical scattering that is consistent with physics.

With the strength σ in (10) decreasing, the maximum defect size moves into Region III, where the Weibull modulus α_{III} also continuously increases, while β_{III}remains to be the constant 2. In the low strength region, clearly the modulus $m_{III}(\sigma)$ increases with the decrease of σ, and accordingly the statistical scattering reduces. The lower strength limit at the right tail of Region III is exactly the asymptotic case discussed in Sub-section 2.1.

In most engineering applications, the slope of the flaw size distribution $P(c)$ varies very slowly with c, and consequently the number of the Weibull modulus is usually small. As validated in practice, for many brittle materials the uni-modal Weibull distribution alone is already sufficient, which corresponds to Region II. In the single CNT case, unlike a macroscopic material specimen, the CNT structure is not described by multiple length scales above that of the atomic lattice. Therefore the chance is expected to be small for the flaw size distribution to be abnormally different from the one in Fig. 1. Consequently the number of the moduli for CNTs in the range of engineering interest is very possibly just 1 or 2.

Prior work on the strength of fiber bundles points to the notion that the flaw size may be fixed within one power law segment but vary from segment to segment. Like CNTs, the strength distribution of a Weibull fiber bundle can also be described as segmented [Harlow and Phoenix (1978); Beyerlein and Phoenix (1996)]. According to the theory of Harlow and Phoenix (1978), the underlying mechanism that gives rise to the segmented distributions is a stress-dependent critical cluster size, where 'size' is expressed in terms of$k$ the number of broken fibers contained in the critical cluster. The high strength extreme tail corresponds to $k = 1$, the second highest segment to $k = 2$, and so on. The theory finds that when the individual bond strength is Weibull-distributed with modulus α, then the exponent of the matching segments is $k\alpha$. It has been demonstrated through subsequent works that this basic idea prevails in describing more complex patterns in two-dimensional and three-dimensional systems [Mahesh, Phoenix, Beyerlein (2002)], with or without a pre-existing flaw [Beyerlein and Phoenix (1997)], and assuming either diffuse or localized load sharing among the bonds [Li and Duxbury (1987)]. In the present context of CNTs, this theory would rationalize that, for instance, the largest flaw size c_{max} was not wholly pre-existing but grew under load via a sequence of new bond failures. As the number of samples tested increases, the likelihood of encountering CNTs that are extremely weak (k is large, Region III) or extremely strong (k is small, Region I) increases. Accordingly, the measured CNT modulus $m = 2 - 3$ [Klein (2007); Yu, Lourie, Dyer, Moloni, Kelly, Ruoff (2000)] could indicate, for instance, that the power law segment corresponding to the average strength has a modulus of say α = 1 and a cluster size of k = 2-3. We will return to this analogy in the next section.

As a closing remark of this section, the duality between Weibull and Gaussian is extended from the asymptotic cases to the non-asymptotic regime, i.e. thanks to universality of the power law approximation (central limit theorem), the Weibull (Gaussian) distribution arises as the natural outcome of the finite size serial (parallel) model independent of the statistics of the elements.

3 Interpretation of CNT Testing Results

In this section, we will first estimate the flaw statistics based on the CNT testing data, and then discuss a major issue present in existing interpretation of CNT bundle testing data.

3.1 Effect of flaw statistics

The expected equilibrium concentration of point defects λ in a pristine CNT is reported to be on the order of 10^{-6} or even lower [Collins (2010)], which fits well the so-called the law of rare events, i.e. all the point defects that occur independently from each other can be modeled as the Poisson process, like the occurrence of mutations in a given sequence of DNA. Note this low concentration also justifies non-interaction of flaws assumed in the fracture model, e.g. Eq. (2). These point defects serve as possible origins to subsequently evolve into nanometer size flaws due to various physical and chemical effects, e.g. oxidation pitting. Therefore the flaw model consists of two steps; the first a Poisson process to produce the number n and locations of point defects, and the second step the evolution of a single defect into a nanometer flaw that contains connected and strongly correlated defects. The second step determines the size distribution of flaws. In connection with earlier fiber models, broad flaw size distributions can lead to segmented strength distributions.

A CNT with diameter D = 20 nm and length L =5 μm (aspect ratio = 250) is chosen as a benchmark, as it is representative of CNTs tested in studies in the literature (Yu, Lourie, Dyer, Moloni, Kelly, Ruoff, 2000). As the CNT contains about $N \sim 10^7$ carbon atoms, with the rate λ = 10^{-6} a quick estimate for the defect free probability shows $P(n=0) = \exp(-10) = 4.54 \times 10^{-5}$, i.e. with such a defect concentration there is little chance of reaching the theoretical strength in tensile testing (Yu, Lourie, Dyer, Moloni, Kelly, Ruoff, 2000) to the three-digit level. In contrast, if we were to reduce the CNT length to 0.5 μm, i.e. reducing the aspect ratio from 250 to 25, the defect free probability drastically increases nearly 10,000 times to$P(n=0) = \exp(-1) = 0.369$. This simple estimate tells there should not be much surprise when a tensile test on a CNT with the length $\sim 0.5\ \mu$m shows a defect free strength at the three-digit level [Demczyk, Wang, Cumings, Hetman, Han, Zettl, Ritchie (2002)].

Based on the results of atomistic simulation [Zhang, Mielke, Khare, Troya, Ruoff, Schatz, Belytschko (2005)], in the following estimate the parameter β in Eq. (2) is taken as 2, and $T/\psi\ \approx 40$ GPa$\sqrt{\text{nm}}$ [Zhang, Mielke, Khare, Troya, Ruoff, Schatz, Belytschko (2005)]. With (2), the critical flaw size c between 1∼6 nm corresponds to the range of strength between 16∼40 GPa, which corresponds to the central part of the sampled distribution [Klein (2007)], apart from the extreme upper and lower tails of the distribution. As data in this core region is considered the most reliable, we use it to estimate the corresponding power law approximation of the flaw distribution $1-P(c) \approx Ac^{-\alpha}$, $c \in [1\text{ nm}, 6\text{ nm}]$. Segments in the lower tail would correspond to larger size flaws and upper tail to smaller ones.

The sampled Weibull modulus in the core range is 2.5. The slope α is estimated to be 1.25 since β =2. The length of the weakest link element is chosen to be 50 nm about the minimum length. Given the mean strength being 30 GPa, with N_e=100 from Eq. (9) the scale parameter is found to be 213 GPa. Further from Eq. (8) we obtain A= 0.0153, i.e.

$$1-P_\ell^c(c) \approx 0.0153c^{-1.25},\ c \in [1\text{ nm}, 6\text{ nm}] \tag{13}$$

With the above result, the true weakest link distribution (9) shows that $P_L(18.5GPa) = 0.2$and $P_L(40.7GPa) = 0.8$, which compare well with the Weibull fitting (Klein, 2007). The estimate (13) tells that 1.53% and 0.163% of CNTs have the maximum flaw more than 1 nm and 6 nm, respectively.

3.2 A comment on back calculation of CNT bundle testing

Since high uncertainty is expected in measurement of both mechanical and geometrical properties of individual CNTs, the CNT bundle testing is considered to be the alternative approach. From the fabrication perspective, the idea of the CNT bundle and yarn also provides an engineering approach to eventually bring the high strength of carbon nanotubes up to macroscopic applications, e.g. [Beyerlein, Porwal, Zhu, Hu, Xu (2009)]. In [Yu, Files, Arepalli, Ruoff (2000)] the strength data for a dozen of bundles of single-walled CNTs (SWCNTs) were reported. The strength of individual CNTs was simply back-calculated as division of the total tensile force by the total wall areas of CNTs at the perimeter of a bundle. This calculation however overlooked the load transfer effect due to heterogeneity of fracture strength of individual CNTs, i.e. the CNTs in a bundle are not broken simultaneously under an identical tensile force, but rather participate in a fracture process involving a non-uniform distribution of tensile force by consecutively transferring the load from a newly broken CNT to its neighbors. The calculation therefore underestimated almost half of the strength of individual CNTs. In other words, the strength reported in [Yu, Files, Arepalli, Ruoff (2000)] actually corresponds to that of a ring of CNTs, but not individual CNTs. The correct procedure to account for the local loading transfer has been described in [Xu, Hu, Beyerlein, Deodatis (2011)]. Below we just provide a lower bound estimate for individual CNTs.

Based on the zeroth-order generalized local load sharing (GLLS) rule [Xu, Hu, Beyerlein, Deodatis (2011)] or equal load sharing rule [Daniels (1945)], the mean strength for a ring of CNTs is given as

$$\bar{\sigma} = \phi \sigma_L \left(\frac{1}{\alpha}\right)^{\frac{1}{\alpha}} \exp\left(-1/\alpha\right) \tag{14}$$

where σ_L is the scale parameter for individual CNTs at the gauge length L, and ϕ the factor to account for the bundle size (number of fibers) effect. For infinitely large bundle sizes, the factor ϕ becomes 1 reducing to Daniels' asymptotic result [Daniels (1945)]. In the finite size case with dozens of CNTs in a ring, the factor ϕ is found to be slightly larger than 1 by using the zeroth-order GLLS simulation [Xu, Hu, Beyerlein, Deodatis (2011)]. According to experimental measurement [Barber, Kaplan-Ashiri, Cohen, Tenne, Wagner (2005)], the ring of CNTs has the Weibull scale parameter 33.9 GPa, and modulus α =2.7, which corresponds to the mean strength 30.1 GPa. The Weibull modulus of individual CNTs certainly is smaller than 2.7, the modulus for the ring of CNTs, due to the upscaling effect as demonstrated in [Xu, Hu, Beyerlein, Deodatis (2011)]. By simply letting $\phi = 1$ and α =2.7 for CNTs, according to Eqn 11 a lower bound estimate for σ_L, the scale parameter of individual CNTs, is found to be 63.0 GPa, which is almost double of the original estimate made in [Yu, Files, Arepalli, Ruoff (2000)]. This brings the mean strength of SWCNTs in [Yu, Files, Arepalli, Ruoff (2000)] from 30.1 GPa up to 56.0 GPa. Considering the diameter of SWCNTs is about 1/10 of those CNTs described in Subsection 3.1, and the gauge length is not more than 50 μm, this increase in strength is consistent with the Poisson distribution as the total number of carbon atoms is reduced.

4 Conclusion

In this note, we formulate a power law approximation to explain the seemingly robustness of Weibull distribution in experimental fitting of the strength of brittle materials. We further propose a generalized Weibull distribution to account for all non-asymptotic cases. In pursuing this theme, published CNT test data are analyzed, and a major issue pertaining to improper interpretation of CNT bundle test data to infer single nanotube properties is discussed in some detail.

While still facing many critical challenges, carbon nanotubes remain the most promising low-dimensional structural components for three-dimensional assembling of the next generation super-strong materials and structures. It is not difficult to envision the statistical characterization developed today will evolve into the future standards for quality control of industrialized high strength CNTs, in a way similar to that routinely practiced in nowadays fiber industry. By justifying the wide applicability of the Weibull distribution to CNTs and other brittle materials such as nanowires, e.g. [He, Xiao, Zhao, Dai, Ke, Zhu (2011)], and clarifying a major issue in the interpretation of CNT bundle testing, we hope this note will provide useful information especially to experimentalists.

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Appendix

$$err(x) = \frac{\left|P_W(\sigma) - P_L^(\sigma)\right|}{P_L^(\sigma)} = \frac{\exp(-N_e x) - (1-x)^{N_e}}{1-(1-x)^{N_e}} \tag{A1}$$

where $x = \left(\frac{\sigma}{\sigma_0}\right)^m$. The maximum of the error function can be found by letting $\frac{\partial err}{\partial x} = 0$, which yields that

$$\exp(N_e x) - x = (1-x)^{1-N_e} \tag{A2}$$

When N_e is large, e.g. bigger than 50, and x is less than 1, $\exp(N_e x) - x$ can be approximated as $\exp(N_e x)$, i.e.

$$\exp(N_e x) \approx (1-x)^{1-N_e} \tag{A3}$$

which has the solution

$$x = 1 + \left(1 - \frac{1}{N_e}\right) ProductLog\left[\frac{N_e}{1-N_e}\exp\left(\frac{N_e}{1-N_e}\right)\right] \tag{A4}$$

where ProductLog gives the principal solution for w in $z = we^w$. Substitution of the solution (A4) into (A1) yields the result shown in Fig. 2. Note the solution (A4) is always less than 1 and the approximation made in (A3) is sufficiently accurate for $N_e > 50$.