Proof Test Methodology for Composites[1]

by

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Summary

The special requirements for proof test of composites are identified based on the underlying failure process of composites. Two proof test methods are developed to eliminate the inevitable weak fiber sites without also causing flaw clustering which weakens the post-proof-test composite. Significant reliability enhancement by these proof test methods has been experimentally demonstrated for composite strength and composite life in tension. This basic proof test methodology is relevant to the certification and acceptance of critical composite structures. It can also be applied to the manufacturing process development to achieve zero-reject for very large composite structures.

Introduction

High performance composites are being specified for load bearing structures in increasing number for land, sea, air and space applications. These applications include man-safe vehicles and hazard containers which demand high safety; inaccessible-after-launch space applications which require high functionality; and very large structures such as ship and submarine hulls which must be manufactured meeting zero reject criterion if they are to be economically feasible. The structural reliability in all these applications requires quantitative reliability estimation and reliability assurance.

For conventional materials such as aluminum or steel, the reliability methodology is frequently experience based. Factors of safety are determined from an extensive data base that have been refined from up to 100 years of structural application experiences. Conversely, for high performance composites, significant improvements in reinforcing fibers and matrix binders are continually being developed . The number of permutations and combinations of fibers and matrixes for different composites is approaching intractable. The very attribute of specifically tailored composite for specific applications also reduces the common reliability data base. Without an

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adequate engineering application data base, reliability of composite must be based on the analytical modeling of its intrinsic failure processes and the experimental confirmation of the model and measurement of the model parameters.

This investigation explores one aspect of composite reliability: the composite reliability which is dominated by the fiber filament tensile failures. Current understanding of the composite failure process in tension is first reviewed and the salient parameters which affect the composite reliability are identified. By combining this physical understanding with the mathematical failure model, two proof test methods are developed to enhance composite reliability. The first proof test method deals with subjecting manufactured articles to preload prior to putting it into service, this is relevant to certification and acceptance methods. The second method deals with pre-loading during processing, this is relevant to manufacturing. The benefits of these two proof test methods are experimentally demonstrated on two different composite systems; one an aramid-epoxy composite, the other a graphite-epoxy composite.

Background on Composite Tensile Failure Mechanism

A rational attempt to improve composite reliability may start with an understanding of its failure process. Through the work of Rosen [1, 2], composite failure process under tension is known to be microscopically sequential starting with failures of the very weak fibers at low load levels. The loads carried by the broken fibers are transferred via the matrix to the neighboring intact fibers, thereby producing a micro-redundancy as illustrated in figure 1a
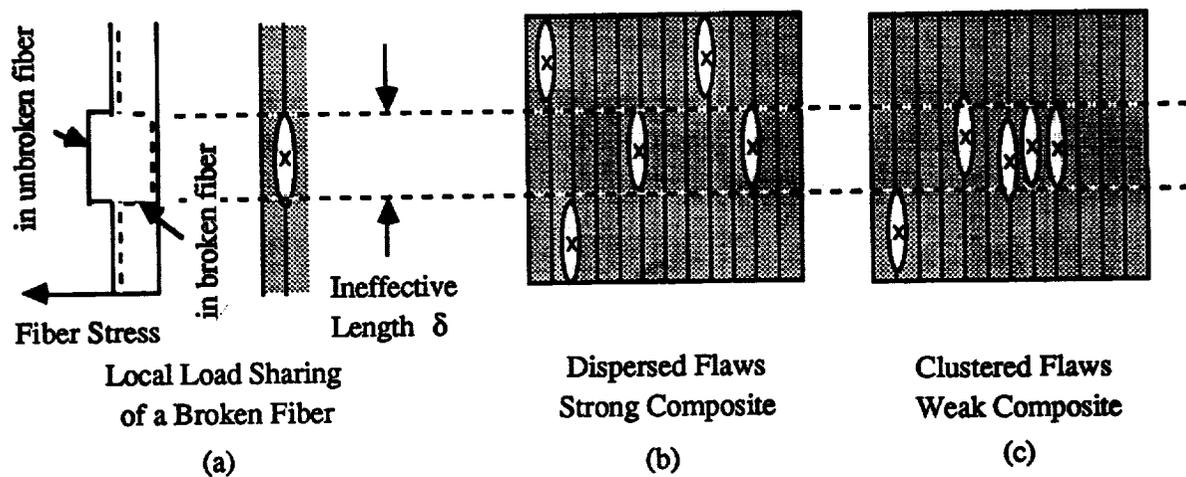

Local Load Sharing
of a Broken Fiber

(a)

Dispersed Flaws
Strong Composite

(b)

Clustered Flaws
Weak Composite

(c)

Figure 1. Local Load Sharing around broken fibers cumulating to flaw clustering .

This process initiates from spatially dispersed sites. As the externally applied load continues to increase, more failure sites are created which leads to clustering and ultimately, catastrophic failure. The longitudinal distance along the fiber required to transfer the stress from one broken fiber to its neighboring unbroken fiber, termed the ineffective length, is estimated by Rosen [1] to be:

$$\delta = \left\{ \left(\left(V_f^{-\frac{1}{2}} - 1 \right) \frac{E_f}{G_m} \right)^{\frac{1}{2}} \cosh^{-1} \left\{ \left[\frac{1 + (1 + \varphi)^2}{2[1 - \varphi]} \right] \right\} d_f \right\} \qquad (1)$$

where:

V_f is the volume fraction of fiber

E_f is the modulus of fiber

G_m is the shear modulus of matrix

φ is the fiber load sharing efficiency; a fractional value below which the fiber is considered to be ineffective

d_f is the fiber diameter

It can be calculated from fiber filament strength statistics that local fiber failure occurs at load levels considerably below the macroscopic catastrophic failure load. For a graphite composite at a typical service stress level, there already exists 3 to 4 fiber failure sites per cm^2 of a single layer of lamina. Even for a moderately sized structure, the number of failure sites add up to millions throughout the structure. The strength of a specific structure ultimately depending on the chanced dispersion or clustering of the inherent failure sites as illustrated in figures 1b and 1c. The chance clustering and the resulting stress concentration from local load sharing have been modeled by Harlow and Phoenix [3,4] using a recursive analysis of the permutations and combinations of the fiber failure sequences and the stress concentrations associated with each configuration. The probability of failure due to nominal far field loads and the local load sharing stress concentrations among adjacent failure sites are partitioned by the ineffective length δ. The Harlow-Phoenix Local Load Sharing model can be used to predict the probability of failure of the composite if the constituent fiber strength statistic and the ineffective length δ are both known. The mathematical structure of this model is of a modified Weibull form and it predicts that statistically weak composite samples typically have a larger number of adjacent break clusters which reduce the macroscopic strength.

It follows immediately that the mechanistic principle for a proof test is to eliminate adjacent breaks, thereby eliminating any substandard weak composite samples. With this understanding of the composite failure process in tension, it is now appropriate to re-examine the proof test as a method for improving composite reliability.

Background on Proof Test

The mechanical proof tests consist of applying a pre-load to a structure prior to putting the structure into service. The proof test load level may be the same or higher than the service load level. The underlying premise of the proof test is that any unacceptably weak structures (in load bearing capacity) are eliminated by the pre-load. The remaining structures which passed the proof test are assumed to have sustained no permanent damage during the pre-load excursion; thus assuring the reliability of those specific structures in subsequent service. Stated in probability terms, all the structures before and after the proof test are idealized to belong to the same population with a probability of failure modeled by :

$F_1(\sigma)$ $\equiv$ Cumulative probability of failure at service conditions $\leq \sigma$

$F_2(\sigma_p)$ $\equiv$ Cumulative probability of failure at proof test conditions $\leq \sigma_p$ (2)

  σ_p $\equiv$ Stress level at proof test

$F_p(\sigma)$ $\equiv$ Cumulative probability of failure after proof test at σ_p

Under the idealized assumption that no permanent strength reducing damage has been incurred during the pre-loading, the post-proof-test population is identical to the pre-proof-test population and the cumulative probability of failure after the proof test is simply conditioned to the probability of survival during the proof test:

$$F_p(\sigma) = \begin{cases} 0, & \sigma < \sigma_p \\[2em] \dfrac{F_1(\sigma) - F_2(\sigma_p)}{1 - F_2(\sigma_p)}, & \sigma \geq \sigma_p \end{cases} \qquad (3)$$

When the condition of the proof test is the same as the service condition, $F_1 = F_2$, equation (3) reduces to the usual definition of conditional probability. In this investigation, we explored the proof test environment for F_2 in order to enhance the post proof test reliability. For the purposes of graphical representation of the effect of the proof test, and without loss of generality, the Weibull distribution is selected to represent the probability of failure function for a composite structure before the proof test:

$$F_i(\sigma) = 1 - \exp\left\{-\left(\frac{\sigma}{\beta_i}\right)^{\alpha_i}\right\} \; ; \quad i=1,2 \qquad (4)$$

The cumulative probability of failure before and after the idealized proof test are shown in figure 2a and repeated in linearized weakest link coordinates in figure 2b

under the transformations:

$$F^* = \ln(-\ln(1 - F))$$
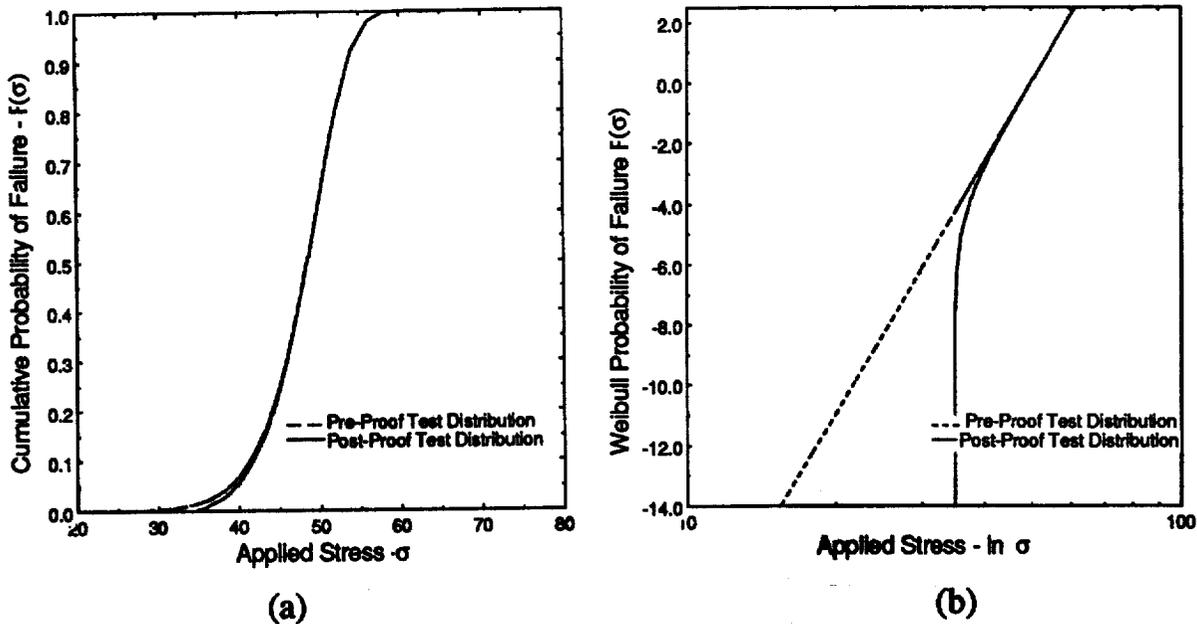$$\sigma^* = \ln \sigma \qquad\qquad (5)$$

Figure 2. Effect of idealized proof test with truncated lower tail.

The effect of an *idealized* proof test is graphically illustrated by noting that at any stress level, the post proof test probability of failure (at high stresses) is always equal to or (at low stresses) less than the pre-proof test probability of failure. The beneficial effects can be observed from either graphical representation. However, the transformed linear representation (figure 2b) has the added advantage that the effect at lower stress levels is visually magnified. This idealized model is applicable to the proof test of a perfectly brittle material where micro-damage, such as flaw growth, is completely absent. In this case, the sample surviving the proof test is guaranteed to be capable of sustaining at least the proof test load level in service. On the other hand, if the idealized no-permanent-damage assumption is *not* true, then the samples which survive the proof test will be weakened. An example is in the of proof testing of a time or history dependent material with flaw growth. This weakening is most severe for samples which have marginally passed the proof test but because of flaw growth, will result in a higher probability of failure following the proof test as illustrated in figures 3a and 3b, again with the transformed representation (figure 3b) clarifying the lower tail effects. Since failure below service stress is the main concern, the weakest link transformation representation is used here to present and discuss the experimental data in order to observe and assess the beneficial or detrimental effects of the proof testing.
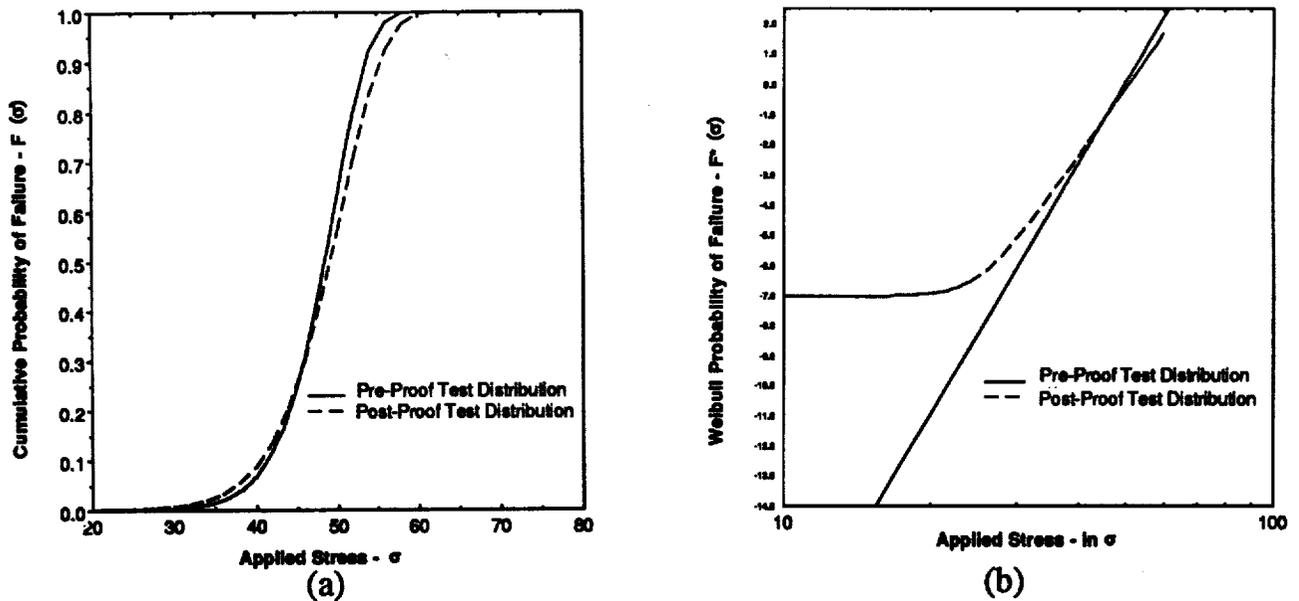
Figure 3. Effect of proof test resulted in damage to lower tail.

The main focus of interpretation of the experimental results herein is whether a particular proof test method is *beneficial,* which results in data *below* the original distribution; or *detrimental,* which results in data *above* the original distribution.

The linearity of experimental data (or lack thereof) is not the main focus; linearity simply suggests that over the range of experimental observations (number of samples tested), the failure process can be represented by the classical Weibull distribution (Eq. 4). In fact, the Local Load Sharing model which best represents the composite failure process is known to be a modified Weibull function which is not linear over a wide probability range. The subject of mathematical modeling of the post proof test distribution will not be discussed.

Proof Test Guideline for Composites

One important result of the local load sharing model is that weak composite samples have an inherently higher number of adjacent fiber breaks (clustered failure sites). It follows immediately that the mechanistic principle for the proof test is to *eliminate adjacent fiber breaks thus eliminating those unacceptable weak composite samples.*

The composite sequential failure process with local fiber failures at low loads directly contradicts the idealization that no permanent damage sites are sustained during proof load. Therefore, conventional proof test methods for homogeneous metals are not applicable for composites. Pre-loading composite articles above service-load levels is sure to cause more local fiber failures while the accompanying stress concentrations induce (otherwise avoidable) additional clustering and weakening of the composite after it has

survived the proof test. Therefore, a beneficial proof test for composites must seek out the weak fibers (whose breakages are inevitable during service) but without creating the accompanying stress concentrations thus avoiding excessive fiber breakages and clustering. The physical parameters which make this condition possible can be inferred from the composite failure model. The Harlow-Phoenix Local Load Sharing model suggests that by increasing the ineffective length, the probability of clusteringis reduced, particularly for the weak (lower tail) composites. It follows that if the ineffective length can be increased during the proof test, then the weak composite will be eliminated while the surviving composites will not be damaged by clustering. This can be physically accomplished by two methods.

Method 1: Proof Test for Cured Composites

This first method applies to a composite that has already been processed and whose matrix has polymerized or hardened. Equation (1) indicates that the ineffective length, δ is inversely proportional to the shear modulus of the matrix. For a polymeric matrix composite, the shear modulus of the matrix is temperature dependent and decreases drastically above T_g (the glass transition temperature). Thus, when the composite is proof tested above its glass transition temperature, the reduction in matrix shear modulus leads to an equivalent (effective) increase of ineffective length, which in turn reduces the stress concentration of the broken fibers. As a result, auto-clustering is averted and only composite samples with inherently clustered weak sites are eliminated by the proof load.

The method is experimentally verified on an aramid-epoxy composite. Composite sample configuration was a matrix impregnated strand with a gauge length of 10 inches. Each sample consisted of a Kevlar 49 bundle with 267 filaments impregnated in a DER332/T403 epoxy/hardener matrix. The epoxy matrix had a glass transition temperature centered at 70° C with full rubbery modulus reached by 100° C.

The effects of proof test carried out at glass transition temperature are explored by examining the life of the composite under sustained load (the stress rupture life). The life data for the same sustained load level (8.29 kg) are used for several proof test conditions. The baseline for comparison is sample life without the pre-loading proof test. This is shown in figure 4 presented in weakest link transformation (Eq.5) with time to fail as the random variable. This graphical format is used because of the exposition of the lower weak tail (short life statistics). The model fitted to the data is the two parameter Weibull distribution with the parameters estimated by the maximum likelihood estimator. It should be noted that there is additional scatter at the lower tail (six points) with a significantly shorter life than that predicted by the two parameter Weibull model.
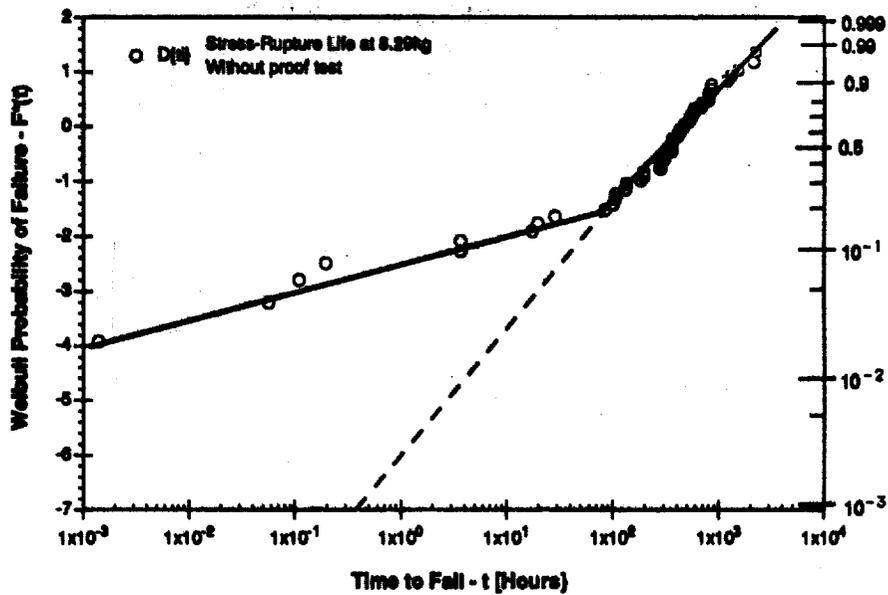
Figure 4. Life of aramid-epoxy composite under sustained load(8.29 kg).

Proof loads at room temperature (23°C) were applied to a second set of samples. Proof load levels were chosen to eliminate 10% of the lower tail. The surviving samples were tested in stress rupture at the same load level (8.29 kg) as the baseline data. The life data are presented in figure 5 with the base line model also presented for comparison. We note that weak lower tail samples recur even after survival of the proof load. This is believed to be caused by those samples with marginal strength above the proof test level but weakened by flaw clustering induced during the proof test which caused the shortening of life under subsequent loading.
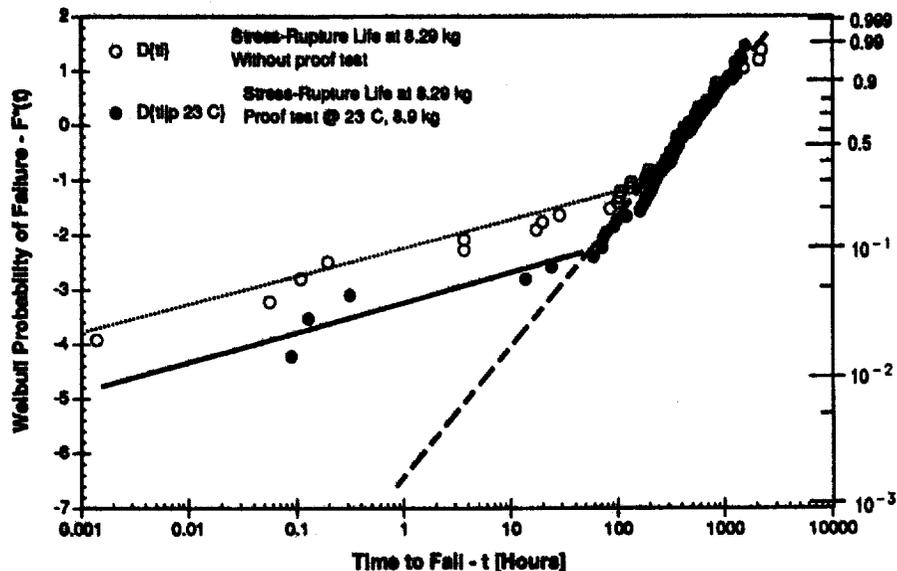

Figure 5. Life of aramid-epoxy composite under sustained load(8.29 kg) after survival of preload (8.93 kg) at 23°C.

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Proof loads were applied to a third set of samples at an elevated temperature (70°) to eliminate 10 % of the lower tail. The surviving samples were slowly cooled back to 23°C then tested in stress rupture. This third set of life data is presented in figure 6.
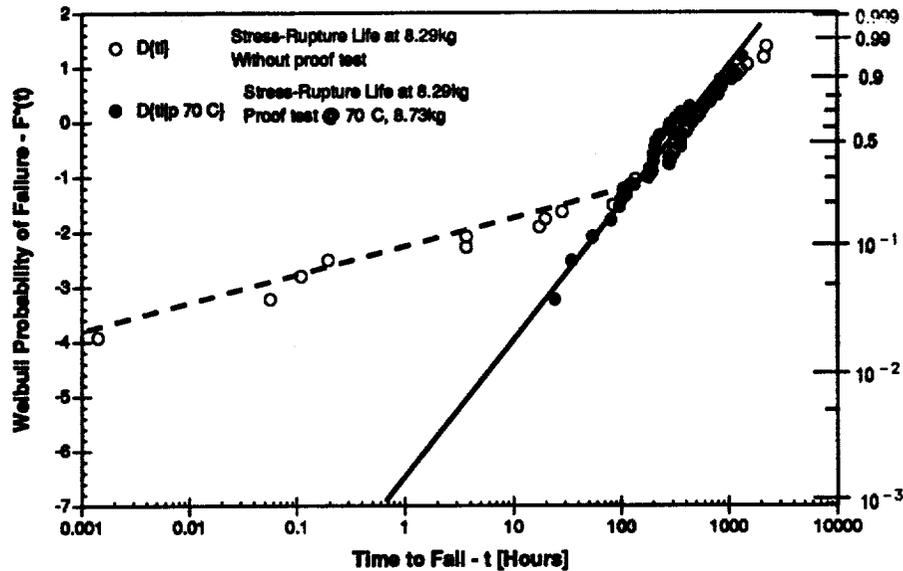

Figure 6. Life of aramid-epoxy composite under sustained load(8.29 kg) after survival of preload (8.73 kg) at 70°C.

Note that the lower tail in life is now totally eliminated. This data substantiates the mechanism that the decrease in matrix shear modulus simultaneously increases the ineffective length and decreases the stress concentration. Both of these parameters minimized flaw clustering thus averting permanent damage during the proof test.
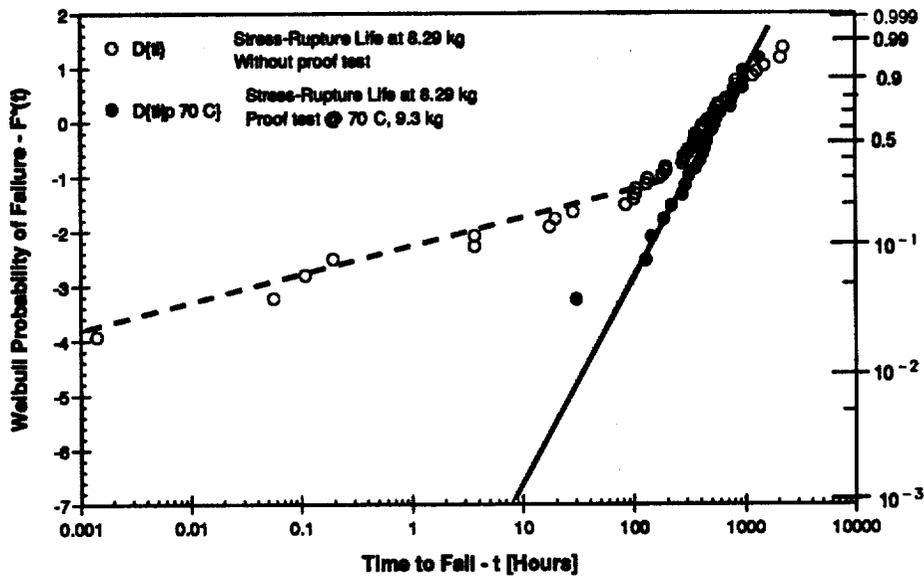

Figure 7. Life of aramid-epoxy composite under sustained load(8.29 kg) after survival of high preload (9.3 kg) at 70°C.

This proof test method is further confirmed on a fourth set of samples where the proof load level was increased to eliminate the lower 50% of the samples at 70°C. The life data are presented in figure 7. We observed that the stress life is further increased as predicted by conditional probability that samples survived elevated temperature proof tests were not damaged.

This proof test method is relevant to the acceptance of composite structures when the level of reliability at a prescribed service condition must be certified.

Method 2 Proof test during processing

This method consists of proof testing the matrix impregnated fiber bundle before the complete polymerization. The physical rational behind this method is that when the matrix is in the liquid state, the composite behaves as a bundle without a matrix, and load sharing stress concentration is absent and therefore, no unnecessary failure sites are created during the proof test. The trade-off is that, in the uncured state, the ineffective length is the same as the gauge length of the pre-load. This was thousands of times longer than the ineffective length for a polymerized epoxy. The large effect length prevents the elimination of *all* the weak fiber sites along the fiber filament and the post proof test benefit is not as complete. However, this can be optimized by proof testing at the matrix partially polymerized state where the ineffective length is decreased. This method has the added benefit that upon curing, the matrix has a measured healing influence on the broken fiber sites.

This proof test method is experimentally verified by performing strength tests on graphite-epoxy samples respectively with and without pre-loading during processing. Samples were of the strand configuration with 10 inch gauge length. AS4 graphite bundles with 3000 filaments were impregnated in a DER332/T403 epoxy/hardener matrix.

Two groups of samples were prepared. One group of samples were allowed to cure without proof load. The second group of samples was allowed to cure for 19 hours at room temperature which brought the epoxy to gel state. These samples were each pre-loaded to 0.9% strain (a level calculated to eliminate the lower 13% of filament flaws at the 10 inch gauge length). After the pre-load, the samples were final cured at 60°C for 16 hours.

These two groups of samples were individually tested in tension until failure. The strength data for the non-preloaded standard sample and the preloaded sample are graphically presented in figure 8.
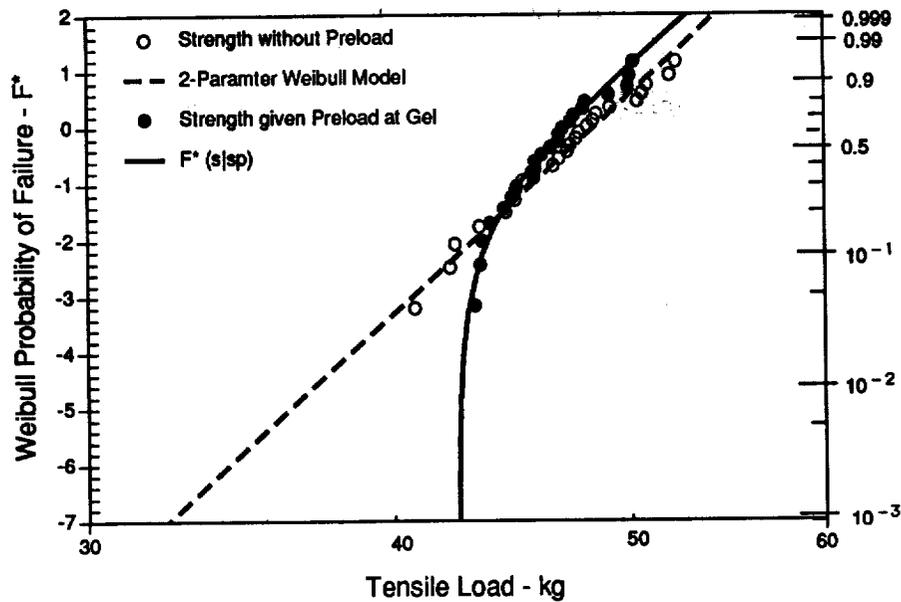
Figure 8. Strength of graphite-epoxy composite verse strength of composite preloaded at gel state during fabrication.

It is clear that the lower tail weak samples were eliminated by the preloading during processing as expected. An unexpected result is that the upper tail is somewhat weaker as compared to the non-preload samples. This could be attributed to fiber misalignment from the dynamic waves of the broken fibers during pre-load. The drawback may be resolved with further development of the processing procedures. In any case, in structural reliability, the upper tail is of relatively minor concern in service conditions. This is further illustrated by the histograms of composite strengths in figure 9. These distribution free histograms indicate that the pre-load before cure composites (shaded histogram) have the lower tail truncated, inferring that the pre-load composite is 100% reliable below the truncation stress level.
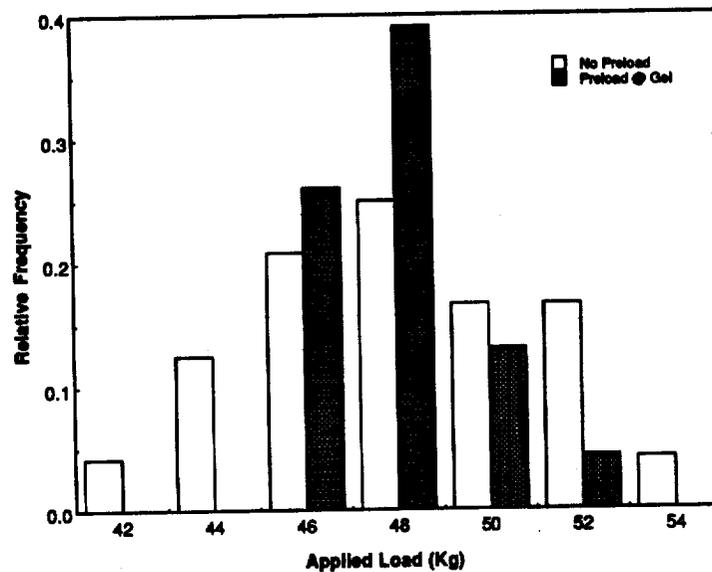

Figure 9. Distribution free strength comparisons of graphite-epoxy composite verse composite preloaded at gel state during fabrication.

This method may be relevant to development of manufacturing processes for very large structures (such as in ship and hulls) and where economic constraints demand zero-reject in manufacturing.

Conclusions

The rationale of applying a proof test to assure composite reliability was examined against the current understanding of the composite failure process in fiber failure dominated tensile failure modes. It was observed that the proof test conditions for composites must be optimized to prevent permanent damage to the samples which survive the proof load. Extensive experimental evidence indicates that proof tests performed in the conventional manner have no beneficial effect, and probably detrimental effects on the composite post-proof test reliability. Two proof test methods were established to prevent permanent damage in terms of excessive fiber breaks and breakage site clustering. The first method involved the proof test of already fabricated composite samples at the glass transition temperature. This method is relevant to certification and acceptance of composite structures. The improvement was experimentally demonstrated on an aramid composite to be over 5 orders of magnitude over an original reliability level of 0.999. The second method consisted of preloading the fiber bundle during fabrication before the complete polymerization of the matrix. This method is relevant to development of zero-reject manufacturing processes. The improvement was experimentally demonstrated on a graphite-epoxy composite to be over 30% at a reliability level of 0.999. At higher reliability levels, the improvement is even more dramatic.

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Materials and Processes Used for
Bonded Repairs of F/A-18 Advanced Composite
Honeycomb Sandwich Structures

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