A Comparative Analysis of Techniques used to Estimate the Mean Recoil Compressive Strength of High Performance Polymers

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Abstract

This study assesses the accuracy of two techniques used to estimate the mean compressive strength of high performance polymers: the Method of Allen and the Moving Average Method. Each method was applied to 10 tests of 80 fibers each and the results of these tests are examined against the two-parameter Weibull model calculated from all the data points collected. The results of these tests have shown that both the Method of Allen and Moving Average Method tend to over-estimate the compressive strength calculated by the Two-Parameter Weibull Model, yet both estimation methods fall in the range of published values (200 – 400 MPa) for the recoil compressive strength of Kevlar – 29 [7]. This over-estimation is contributed each method's assumed linearity of the recoil compressive test data. A statistical analysis of the results was completed and shows with a 95% confidence level that there is no statistical significant difference between the two methods. Although the Method of Allen produces the same estimated mean recoil compressive strengths as the Moving Average statistically, problems associated within the testing procedure for Method of Allen itself leads to the conclusion that Moving Average Method is the better technique for the estimation of the mean recoil compressive strength.

Introduction

High-performance polymers have extraordinary tensile strengths that enable them to be used in a variety of high performance applications. The nearly perfect axial orientation that is formed during the dry-jet wet spinning of high-performance polymers provides the fibers with exceptional tensile strength [1]. With this spinning technique comes a dramatic reduction in the fiber's ability to distribute a compressive load [2]. For example, Kevlar (poly p-phenylene terephthalamide or PPTA) fiber-reinforced composites typically possess compressive strengths that are more than five times less than their tensile strengths [3-5]. Figure 1 depicts the repeat unit of Kevlar. The comparatively poor compressive strength of high-performance polymers severely limits their use in structural applications where the fibers may be subjected to compressive forces.

ASTM [6] has established standards for the compressive testing of composites, but these tests involve many steps, large quantities of fiber, and are influenced by the quality of bonding between the fiber and matrix. Indirect measurements are then advantageous since the majority of high performance polymers are only available as fibers and as such cannot be directly measured for their compressive strength easily. There are several indirect methods proposed to measure the compressive strength of high performance polymers. Bending techniques (ASTM D 3375-79, ASTM D 695, ASTM D 3410, ASTM M 695) are employed to estimate a fibers compressive strength [5,13-16]. While the bend test does provide a comparison in results to the composite test, the compressive strength found is generally much higher, by two or three times, to the compressive strength found by the composite test. Another testing techniques, the single filament composite, yields better agreement to the composite test but requires a tedious procedure that limits its usefulness [17-19]. The recoil compressive test was a technique found to generate results that closely agreed with data from the Composite Test.

Allen [8] proved that, in the recoil compressive test, when a fiber is broken or severed while under a tensile load a snap-back or recoil wave travels through the lengths of both halves of the fiber and causes compressive forces on the fiber equal to the tensile load that was placed upon it when it was broken or severed. This analysis involves making several assumptions about the fiber [8]: (1) the fiber obeys Hook's law for linearly elastic materials, (2) is rigidly clamped at each end, (3) has no initial velocity, and (4) has a uniform initial stress along its length at failure. Such a test yields two data points for every one fiber. The ends of the fiber are examined and then assessed for failures and survivals of the recoil compressive wave, which are assigned the binary numbers “0” and “1” respectfully. Figure 2 depicts recoil compressive failures and survivals and the binary system assigned to represent each. Unlike tensile testing, when the recoil compressive tests is performed, an exact compressive strength of a filament cannot be directly determined. Because the compressive failure is artificially induced by cutting the fiber, the resulting recoil compressive wave may be significantly above or significantly below the intrinsic compressive strength of the fiber. If the fiber ends experience a compressive failure, it is difficult to determine at what level did the stress placed upon the fiber exceed the intrinsic compressive strength of that fiber. To assess an indirect measurement technique like the recoil compressive test, an indirect method of determining the mean compressive strength of high performance polymers must be applied to express the data. Several methods have been proposed to evaluate the data collected from recoil compressive tests.

One method (The Method of Allen) used by Allen [8], Wang et al [9], and Crasto and Kumar [7] arranges the data in ascending order with respect to the stress applied. Two points are then identified, the point at which the lowest failures are recorded and the point at which the highest survivals are recorded, and averaged to estimate the mean compressive strength. This method, currently used by industry, does present several inherent errors associated with the determination the compressive strength. One potential problem that exists is that the method computes the mean recoil compressive strength of a batch of fibers using only two data points, while discarding the rest of the data set. The dangers of this method then are twofold: since the next test performed could significantly impact the computed mean, it is impossible to know when a sufficient number of results have been collected the use of two extreme points makes the result highly vulnerable to outliers.

Newell and Gustafson [10] suggested an improved method of assessing the compressive strength of a batch of fibers from data collected by the recoil compressive test. This method seeks to eliminate the potential problems of data loss present with the Method of Allen by using results of the test conducted beforehand to determine the stress level of the upcoming test. With the Moving Average Method, an original stress limit is set and tested and the fibers are examined after induced failure. If both halves of the fiber survive the test, a stress of 20 MPa (approx. 5% of mean recoil compressive strength) higher is tested on the subsequent fiber; if one half of the fiber passes and the other half fails, the original stress load is retested on the next fiber; and if both halves of the fibers fail, the stress is reduced by 20 MPa. Subsequent tests follow accordingly. After testing a number of fibers, the data points begin to oscillate around a stress level that is close to the mean compressive stress of the batch. In order to eliminate the error associated with the original stress limit, an average of the final thirty data points collected represents the mean recoil compressive strength. Simulations presented by Newell et al. [10] show a clear advantage for the Moving Average over the Method of Allen in predicting the mean compressive strength of high performance polymers.

Since both the Method of Allen and Moving Average are only estimations of the mean compressive strength of high performance polymers, the accuracy of each of these methods estimations need to be examined. Before an assessment of accuracy for either the Method of Allen or the Moving Average can occur, an accurate measurement of the true mean compressive strength must be performed. Newell et al [11] proposed that a rigorous mathematical model, the Weibull Model, should be used to accurately express the mean compressive strength of high performance polymers. Both the two and four parameter models were examined, with the four-parameter model showing little improvement is accuracy over the two-parameter model. The two-parameter model is shown below:

$$F = 1 - \exp[-L(\frac{\sigma}{\sigma_O})^m] \quad (1)$$

Deconvolution of the four-parameter model showed that a single mode of failure dominated the failure of high performance polymers in the compressive mode. This agreed with McGarry and Moalli [20] who analyzed the compressive failure of PPTA using scanning electron microscopy and found that the primary mode of compressive failure in both PBO and PPTA was a "kind band" or jog phenomena. Using the two-parameter, Newell et al [11] determined that the mean compressive strength for a batch of Kevlar-29 fibers was 285 MPa, which is within range of the accepted published values of 200 – 400 MPa [7] for recoil compressive strength for Kevlar – 29. For this study, the two-parameter Weibull model will then be used as a basis of comparison with the data that is collected using the Moving Average method and Method of Allen.

Experimental

Fiber samples of Kevlar-29 were mounted on paper tensile testing tabs and secured with Epoxy 220. Fiber diameters were determined by laser diffraction. The tabs were then secured in the locking grips of a modified Instron Ultimate Testing Machine (Figure 3). A total of ten tests will be run using the two different estimation methods with 80 fibers tested per run. For the first estimation method, the Method of Allen, random stresses ranging from 100 to 800 MPa were formulated using a randomizer program and tested. For the Moving Average, varying starting points were chosen between the values of 100 and 800 MPa. In each test, after a static tensile load was applied to the fiber, the fiber was cut in the center using sharpened surgical scissors. The filament pieces were examined under a magnifying lens for classification as survivals (1) or failures (0). These results were then recorded and analyzed for their estimated recoil compressive strength.

Results and Discussion

Recoil compressive tests were run using both the Method of Allen and Moving Average methods of estimating the mean compressive strength. Each of these tests were conducted over 80 fibers to ensure a fully developed analysis of the two estimation methods. To gauge the progress of each method's ability to converge to the mean compressive strength found using the two-parameter Weibull Model, estimations of the compressive strength were taken at fibers 10, 25, 50, and 80 of each test with the results shown in Table 1.

A two-parameter model was obtained for the 3039 data points tested. The experimental failure data were classified into twelve bins with a stress range of 65 MPa each, as shown in Figure 4. Once the mean average stress and experimental frequency of failure were found for each bin, Weibull parameters were linearly regressed by plotting $\ln[\ln(\frac{1}{1-F})]$ versus $\ln(\sigma)$ as shown in Figure 5 (the two Weibull parameters determined are shown in Table 2). To determine the mean compressive strength using the Two-Parameter Weibull Model, a first moment over zero moment analysis was used. The mean compressive strength determined from the aforementioned data points was found to be 256 MPa.

A statistical analysis was performed on the data to determine any significant differences between the means for the Method of Allen and Moving Average Method. An ANOVA single factor analysis was performed. The results of this analysis show that there were no statistical differences between the two tests means within 95% confidence. The ANOVA table, Table 3, shows there were no statistical differences between the two tests (the p-value was above the critical value of 0.05). A multiple range test, Table 4, was performed between the two groups as well and both were found to be in the same homogeneous group. Figure 6 shows the comparison of the means for the two methods with standard deviations, which also shows that there is no difference between the two methods. T-tests were performed to assess the accuracy of both tests to the accepted mean found using the two-parameter Weibull model. These results are shown in Table 5. It is clear that both methods show a significant difference from the critical t value for the 95% confidence level. Both methods make a critical assumption in order to estimate the mean compressive strength for a batch of high performance fibers. The Method of Allen averages the highest survival and the lowest failure; thus assuming that the data collected using the recoil compressive test is linear. This also holds true for the Moving Average Method: the last thirty fibers test are averaged together to determine the mean compressive strength for that particular test. This assumed linearity is not done in the Weibull Model since this model fits the line from the data itself. The data, collected into a histogram as seem in Figure 4, is clearly not linear and therefore error is associated into each methods estimation of the mean compressive strength. Even so, the mean compressive strengths found by the estimation techniques do fall within the range of accepted published values for the mean compressive strength of Kevlar-29. [7]

The result using the Moving Average to estimate the mean recoil compressive strength for all ten tests is 326 $\pm$ 48.7 MPa. Starting from a various stresses, the Moving Average was able to complete each of the tests to where the stresses were oscillating between two points with the resultant estimated compressive strengths demonstrating this pattern. (Shown in Table 1) This trend is represented in Figure 7, where each line represents a single test using the Moving Average. Represented by the graph, regardless of the starting point, as the stress applied to the fiber is increased, decreased, or kept constant according to the preceding test, the slope of each line decreases until it is oscillating between two points, creating essentially no slope. Of particular note was the ability of the Moving Average to recover from apparent outliers in the data (see Figure 7) and still converge in the range of accepted compressive strengths. This would have proven to be a problem for the Method of Allen since the point would have had to count in the results. It is for this reason, and the fact that there was a definite conclusion to the test using the Moving Average Method as compared to the Method of Allen that the Moving Average would be the preferred test to use when estimating the mean recoil compressive strength of high performance fibers.

A look at the Method of Allen test results give further proof that the Moving Average is the better of the two estimation techniques. The results of the Method of Allen for the estimation of the mean recoil compressive strength for all the tests performed was calculated to be 367 $\pm$ 41.5 MPa. This value is similar to that calculated by the Moving Average in that the method over-estimates the mean compressive strength estimated by the Two-parameter Weibull Model. The trend for each test done using the Method of Allen is represented in Figure 8. As seen in this graph, as a new highest survival or lowest failure is recorded during a test, the test line suddenly jumps to the new estimated average strength. This "jumping" of the test line leads to a large inconsistency in when the Method of Allen determines the mean recoil compressive strength and when the test is concluded. (See Figure 8) Some tests of the Method of Allen come to the estimated strength within the first 25 fibers while other tests do not come to a final value until late in test run. This tends to solidify the claim that a major problem faced when using the Method of Allen is in the determination of a finite number of test fibers needed to accurately estimate the compressive strength.

Conclusions

This study shows that, statistically, both the Method of Allen and Moving Average Method are the same. However, due to the assumed linearity by both methods, they tend to over-estimate the compressive strength calculated by the Two-Parameter Weibull Model. Both estimation methods do fall into the range of published values (200 – 400 MPa) for the recoil compressive strength of Kevlar – 29 [7], and therefore remain acceptable estimation techniques for determining the mean compressive strength of high performance polymers. It is then the conclusion of this study that the Moving Average Method does provides a more confident estimation of the mean recoil compressive strength over the Method of Allen by eliminating most problems associated with the Method of Allen itself; mainly by the convergence of the Moving Average to oscillations between two points and the clear conclusion to a test run.

References

1. Black W B 1980 *Annual Review of Materials Science* **10** 311
2. Tanner D, Fitzgerald J A and Phillips B R 1989 *Advanced Materials* **5** 151
3. Wilfong R E and Zimmerman J 1977 Journal of *Appl. Polym. Sci., Appl. Polym. Symp.* **31** (1) 1
4. Allen S R 1983 *Mechanical and Morphological Correlations in Poly p-Phenylene Benzobisthiazole Fibers* PhD Dissertation, University of Massachusetts, Amherst.
5. Dobb M G, Johnson D J and Saville B P 1981 *Polymer* **22** 960
6. ASTM Standards D695 and D3410
7. Kumar S and Helminiak T E 1990 *SAMPE Journal* **26**(2) 51
8. Allen S R 1987 Journal of Material Science **22** 853
9. Wang C S, Bai S J and Rice B P 1989 *Proceedings of the American Chemical Society, Division of Polymeric Materials: Science and Engineering* **61** 550
10. Newell J A and Gustafson J M 2001 *High Performance Polymers* **13** 251
11. Newell J A, Kurzeja T, Spence M J, and Lynch M 2002 *High Performance Polymers* **14** 425
12. Hayes G J 1993 *Analysis of the Single Filament Recoil Test* PhD Dissertation, Clemson University
13. Schoppee M M and Skelton J 1974 *Tex. Res. J.* **44** 968
14. Greenwood J H and Rose P G 1974 *Journal of Material Science* **9** 1809
15. Smith S C, Lau E K, and Backer S 1978 *Tex Res J* **48** 104
16. Jones W R and Johnson J W 1971 *Carbon* **9** 645
17. Hawthorne H M and Teghtsoonian E 1975 *Journal of Material Science* **10** 41
18. DeTeresa S J, Allen S R, Farris R J, and Porter R S 1984 *Journal of Material Science* **19** 57

19. DeTeresa S J 1985 “The Axial Compressive Strength of High Performance Fibers”, PhD Dissertation, University of Massachusetts

20. McGarry F J and Moalli J E 1991 *Polymers* **32** 1811

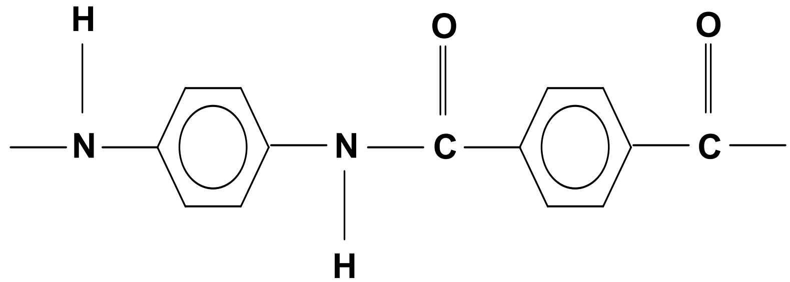

Figure 1: Repeat Unit of Kevlar

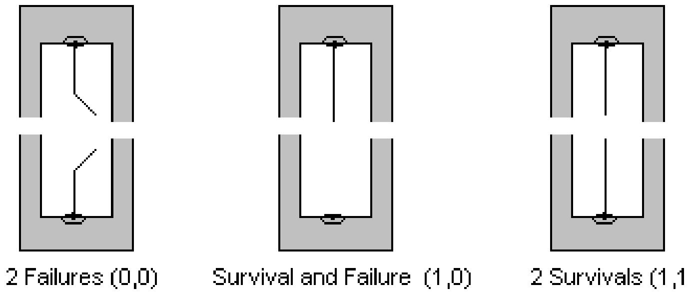

Figure 2: Potential Results of the Single Filament Recoil (Recoil Compressive) Test

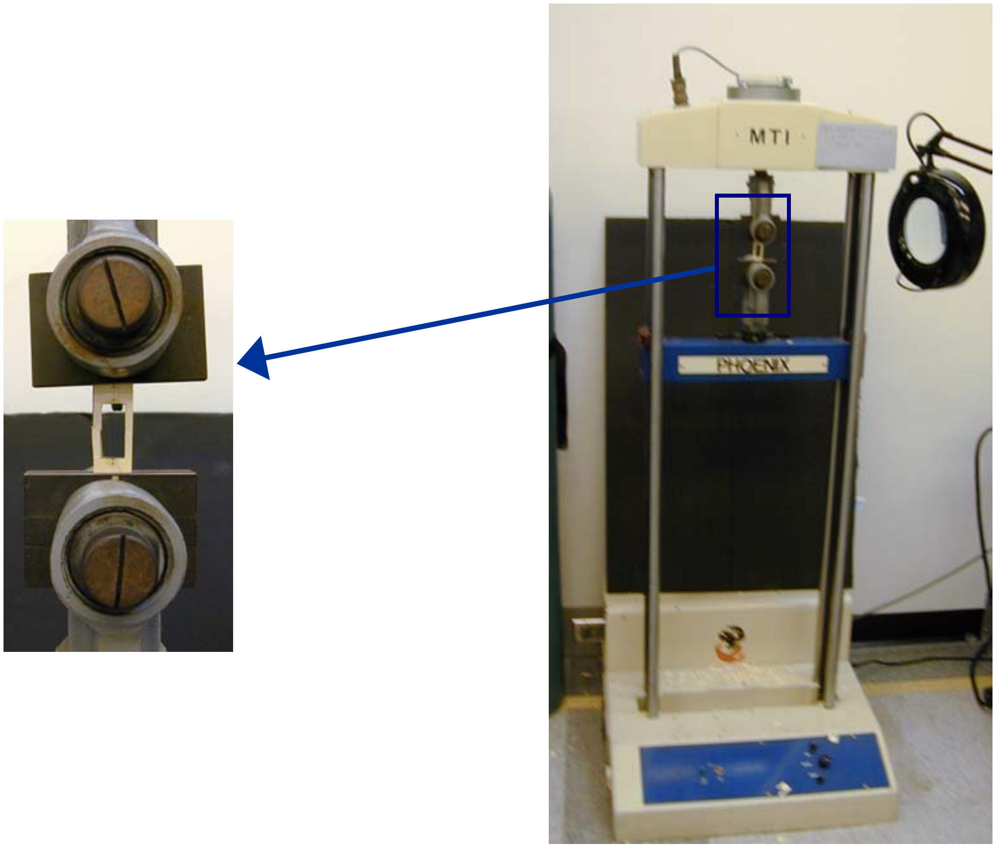

Figure 3: Pictures of Modified Instron Universal Testing Machine with a close up of the placement of the testing tab in the hold clamps

	Moving Average				Method Of Allen			
	Estimated Mean Compressive Strength (MPa)				Estimated Mean Compressive Strength (MPa)			
Test #	**10**	**25**	**50**	**80**	**10**	**25**	**50**	**80**
1	380	328	237	305	137	341	341	341
2	235	248	340	477	313	211	299	406
3	811	733	510	385	266	466	466	466
4	390	412	395	370	undefined	317	326	326
5	248	252	294	307	234	237	270	311
6	528	457	373	288	401	401	326	305
7	145	203	305	330	370	352	339	322
8	398	357	296	260	457	457	454	454
9	625	542	328	266	undefined	332	367	367
10	211	226	258	269	313	312	418	374
Average	397	376	334	326	311	342	360	367

Table 1: Estimated Mean Compressive Strength Data for Different Test Runs (True Mean = 256 MPa)

(* The undefined estimated mean is referring to the fact that after ten fibers tested, there were no double passes or double failures to predict an average mean over that range of fibers using the Method of Allen)

Two Parameter	
m	1.0599
σ_O	1.266293
σ_{AVE}	**256**

Table 2: Two-Parameter Weibull Model Data

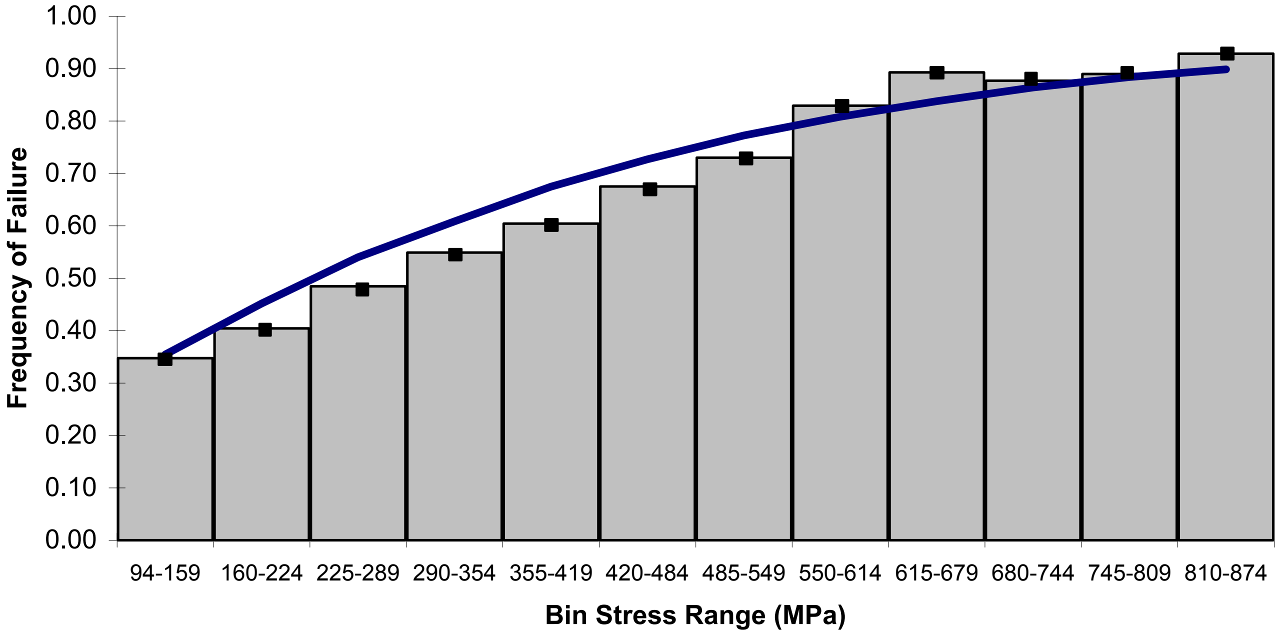

Figure 4: Histogram of Experimental Failure Frequency vs. Applied Stress with Two Parameter Model super-imposed

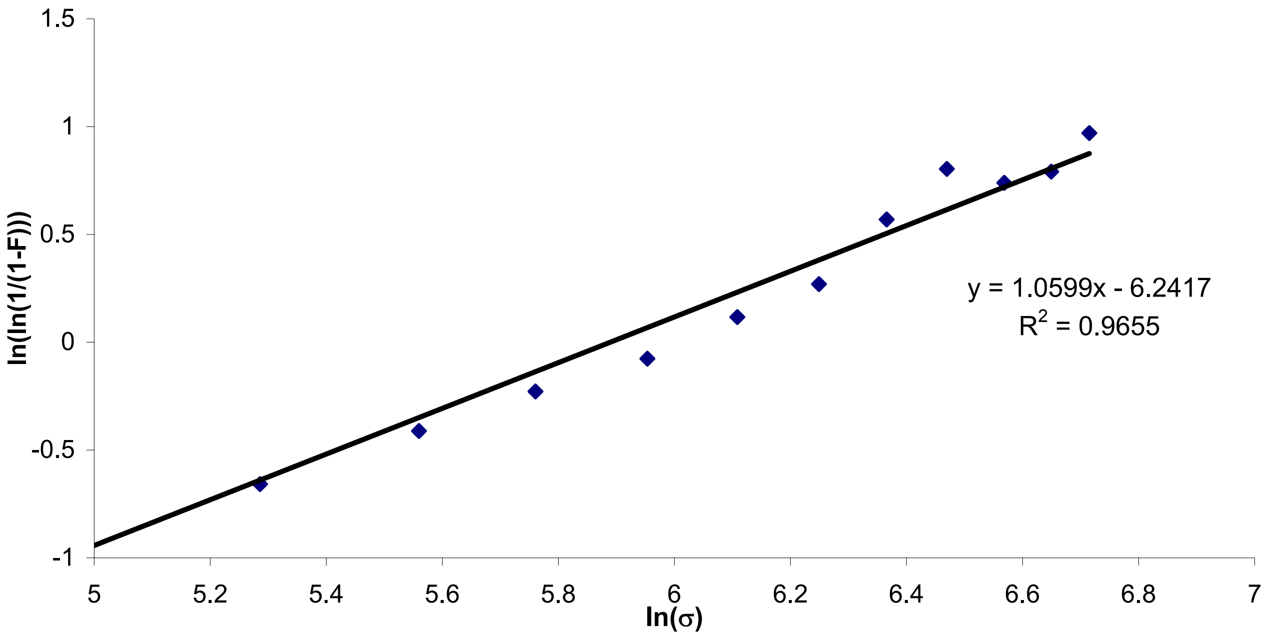

Figure 5: Linearly Regressed Two-Parameter Model

ANOVA Table for Stress by Esti_Metho

Analysis of Variance

Source	Sum of Squares	Df	Mean Square	F-Ratio	P-Value
Between groups	8611.25	1	8611.25	2.15	0.1595
Within groups	71985.7	18	3999.21		
Total (Corr.)	80596.9	19			

Table 3: ANOVA table constructed using STATGRAPHICS statistical program

```
Multiple Range Tests for Stress by Esti_Metho

--------------------------------------------------------------------------------
Method: 95.0 percent LSD
Esti_Metho     Count     Mean              Homogeneous Groups
--------------------------------------------------------------------------------
MA             10        325.7             X
MOA            10        367.2             X
--------------------------------------------------------------------------------
Contrast                                   Difference          +/-  Limits
--------------------------------------------------------------------------------
MA - MOA                                   -41.5               59.4173
--------------------------------------------------------------------------------
* denotes a statistically significant difference.
```

Table 4: Multiple Range Test Analysis of Method of Allen (MOA) and Moving Average (MA) Methods

Figure 6: Means Comparison of the Method of Allen (MOA) and Moving Average (MA)
[At 95% Confidence Level using Least Significant Difference Method]

	Moving Average	Method of Allen
T_{calc}	3.258	6.089
T_{crit}	2.262	2.262

Table 5: T-Test for Accuracy of Each Method to the Accepted Mean

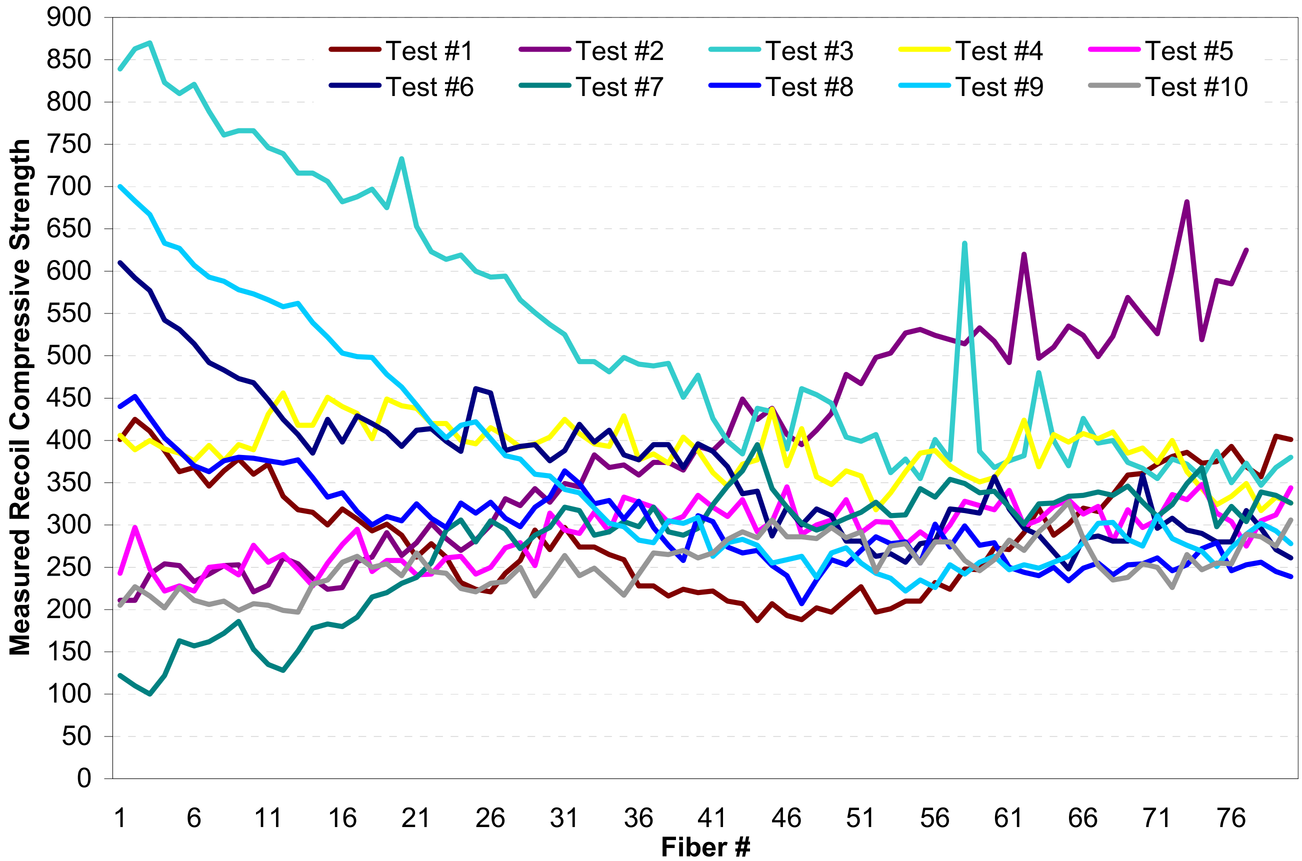

Figure 7: Representative of the Average Compressive Strength Throughout Each Test Run for the Moving Average

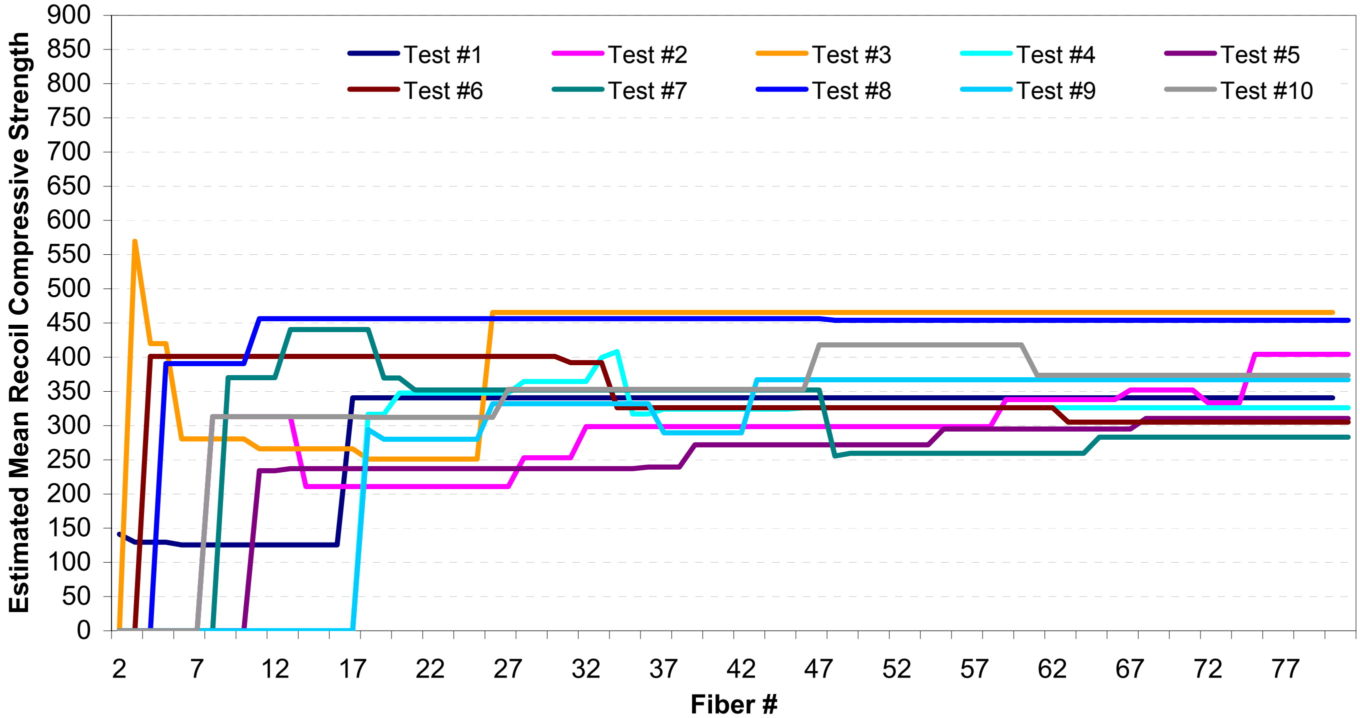

Figure 8: Representative of the Average Compressive Strength Throughout Each Test Run for the Method of Allen