AN ANALYSIS PROCEDURE FOR QUANTIFYING THE FRAGMENTATION BEHAVIOR OF 2-D MULTI-FIBER ARRAYS

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ABSTRACT

With composite materials being targeted for use in structural applications, concerns have increased about their long-term structural integrity, since conventional sub-critical flaw detection methodologies are generally not effective for composites. Furthermore, concerns about composite failure models that are typically used to predict failure behavior of composites in lieu of testing have also increased due to the 1995 research of Li *et al*. and the 2004 research of Kim *et al*. These research results employing micromechanics tests on multi-fiber microcomposites indicate that the underlying *shear-lag* models typically used to predict composite failure behavior show an increase in fiber-flaw density along the length of a composite fiber rather than the experimentally observed decrease. More recently Kim *et al.* have shown that the multiple fiber fracture process observed in a single fiber composite can be captured by statistical models based on lifetime analyses. The output parameters from these analyses may provide a basis for quantifying the fiber failure process in different composite materials. To test the validity of this method, the lifetime analyses approach is extended in this report to the fiber-fracture process in multi-fiber microcomposites.

KEY WORDS: GLASS FIBER COMPOSITES, FAILURE ANALYSIS/FAILURE ASSESSMENT, TESTING/EVALUATION

1. INTRODUCTION

Interest in the use of micromechanics to investigate complex microdamage events that occur in fibrous composites, such as fiber breakage, interface decohesion, matrix failure, critical flaw nucleation, etc., was revived by Wagner and Steenbakkers [1] who described a geometric-based methodology for preparing 2-D multi-fiber microcomposite test specimens. This research extended the often cited research of Rosen *et al.* [2] who investigated 2-D multi-fiber microcomposites composed of 88.9 µm diameter E-glass fibers embedded in epoxy resin, to the investigation of Kevlar and E-glass fibers whose diameters are comparable to those found in fibrous composites. Research by Li *et al.* [3] on 2-D multi-fiber microcomposites using 15 µm Nicalon silicon carbide fibers, also a non-standard fiber for fibrous composite manufacture, indicated that the physics of the repeated fiber fracture process predicted by the van Dyke and Hedgepeth [4] and Zweben [5] shear-lag models is inconsistent with their experimental micromechanics results. These researchers observed, in contrast to the model prediction, that the break density along the length of a fiber in a 2-D microcomposite as the strain is increased decreases as the inter-fiber distance decreases. These results were subsequently verified by Kim *et al.* [6] who tested microcomposite specimens composed of a 2-D E-glass multi-fiber array embedded in an epoxy matrix between two single E-glass fibers (i.e., a combinatorial microcomposite) (see Figure 1). To facilitate data reduction and to control the deformation rate Holmes *et al.* used an automated tensile testing machine with archival capability [7]. Since the van Dyke and Hedgepeth, and Zweben shear-lag models are often used in composite failure models, these results suggest that the physics of the composite failure process may be incorrectly predicted by these models.

Furthermore, these results indicate that failure initiation in fibrous composites is not due to an increase in break density along the length of the fibers leading to an increased probability of generating a critical flaw – as predicted by the above shear-lag model, but due to the fiber-fiber interaction process resulting in the alignment of fiber breaks in adjacent fibers that results from the increasing magnitude of the stress concentration factor (SCF) as the inter-fiber distance is decreased. This observation is supported by the extensive research of Young *et al.* [8-12] on 2-D carbon fiber microcomposites where Raman spectroscopy was used to quantify the magnitude of the SCF on the interacting fibers at various inter-fiber spacings. These researchers also observed that none of the existing micromechanics shear-lag models accurately predicted the maximum SCF with inter-fiber spacing that was experimentally observed in the Raman experiments.

In an attempt to understand better the statistics of the repeated fiber fracture process and its impact on the fragment length distribution in single-fiber microcomposites, Kim *et al.* [13] investigated the repeated fracture process in single-fiber microcomposites composed of E-glass fibers where the extent of debonding can be quantified and minimized by controlling the adhesion strength between the matrix and the fiber [14]. These researchers found for the case of minimal debonding that, above a critical fiber break density, the fiber breaks along the length of the embedded fiber are uniformly spaced. From uniform spacing theory, these results indicate that any agreements researchers have observed between the fragment length distributions at saturation and distributions such as the Weibull, shifted exponential, and log-normal may be fortuitous or approximate at best [13].

For breaks that are uniformly spaced along the length of a fiber, their locations are readily predicted from spacing statistics. Furthermore, since the predicted locations are dependent only on the number of fiber breaks within the finite interval, simulations have shown that a coordinated fracture pattern can be obtained between independently fracture fibers as long as the break density along each fiber is the same and the spacings are uniform. These results suggest that statistics analysis based on the uniform distribution may be useful in predicting fiber fracture positions inside composites if a parameter can be found that captures the fiber break coordination that occurs prior to the establishment of the uniform distribution of fiber breaks. In this paper, experimental test results from a 2-D combinatorial microcomposite composed of E-glass fibers are compared with theoretical predictions to determine how fiber-fiber interactions alter the evolution of the fiber break process in multi-fiber arrays. The results will provide insight into how fiber break locations evolve when the fibers do not fracture independently. In addition, since this type of specimen simultaneously tests the repeated fracture process in two single fibers and in a multi-fiber array, variability in the experimental results caused by the testing of individual test specimens and testing rate are minimized.

2. EXPERIMENTAL

E-glass fibers sized with 3-aminopropyl triethoxysilane (A-1100) were used in this experiment. For the matrix[1], resin mixture of bisphenol-A diglycidyl ether (DGEBA, Epon 828, Shell Co.), diglycidyl ether of 1,4-butanediol (RD-2, Ciba-Geigy), and 1,3-phenylene diamine (m-PDA, Fluka Chemical Co.) was used in a 100:25.1:20.6 mass fraction ratio. Figure 1 shows the schematic of a 2-D combinatorial microcomposite test specimen that consists of a 2-D multi-fiber array in the middle of the specimen and two single fibers at both sides from the center. The 2-D multi fiber array in the middle of specimen will be termed "the cluster fibers". The detailed experimental procedure to fabricate microcomposites with 2-D multi fiber array is available in previous publications [6,15]. Using an automatic loading device, tensile load was applied to the fragmentation test specimen by sequential increments (step strains) of about 0.085 mm at a loading rate of 0.085 mm/min. To achieve a strain of greater than 8 %, 35 steps were performed. The time interval between loading steps was either 10 min or 1 h. Interval censored data was obtained by taking an image of the specimen after each loading step using a 25 magnification digital camera. The observed fragmentation images for each loading step were analyzed by the image analysis software for quantifying a fiber break location.

3. RESULTS AND DISCUSSION

3.1 Evolution of fiber breaks. The evolution of a fiber break process of the cluster fibers in the micro composites as a function of the tensile loading steps is shown in

Figure 2. Fiber breaks occur at 3 % of elongation and increase sharply up to 4% to 5% strain. Then the fiber break evolution process slows around 5 % to 6 % and reaches saturation at approximately 7 % strain. Therefore, the break evolution process in each fiber is consistent with the break evolution process that occurs in the much reported single fiber fragmentation test. In contrast to the approximately 38 breaks observed in the cluster, 51 and 53 breaks were found in the two single-fiber specimens in the 2-D combinatorial microcomposite. These results are consistent with the previously cited research results of Phoenix and Beyerlein, thereby suggesting by the shear-lag models a reduction in the fiber-matrix interfacial shear strength (IFSS).

As an aside, the break density resulting from the 2-D combinatorial microcomposite suggests that the reduction of approximately 20 % in the number of breaks between the cluster and single fibers is not due to a change of molecular adhesion at the fiber-matrix interface. This reinforces the assumption that the fiber-matrix IFSS, as measured by embedded fragmentation tests, is not a true measure of the molecular adhesion at the fiber-matrix interface. This assertion is based on the observation that even though the spacings between the fibers in the cluster are approximately 15 μm, the epoxy monomers that undergo the curing reaction are approximately 2.5 nm in length. Results from Arayasantiparb *et al.* [16] suggest an upper bound on the thickness of the interphase region where the molecular reactions are perturbed by the presence of the fiber at

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[1] Certain commercial materials and equipment are identified in this paper to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply necessarily that the product is the best available for the purpose

approximately 100 nm or 40 epoxy repeat units. These results suggest that in the matrix region between the clusters, the interphase regions on each fiber surface do not overlap or interact during the curing reaction that establishes the level of molecular adhesion in the fiber-matrix interphase region.

3.2 Distribution of fiber break locations. Consistent with data analyzed from the fracture of single-fiber test specimens [13], the spatially ordered fiber break locations were fitted by probability plotting to test for goodness-of-fit to the uniform distribution (P) using the following equations for theoretical predictions of uniform order statistics

$$P_1 = 1 - 0.5^{\frac{1}{n}},$$

$$P_i = \frac{(i - 0.3175)}{(n + 0.365)}, \qquad i = 2,3,\ldots,n-1, \text{ where n is the sample size (here: number of fiber breaks).}$$

Figure 3 graphically represents these calculations on the fiber break locations at saturation in the cluster fibers, with the correlation coefficients for each fit and the average fragment length in each cluster fiber shown in Table 1. These data indicate that the breaks in the cluster fiber, like the single fibers, are extremely uniform with correlation coefficients of greater than 0.999. Analysis of variance (ANOVA) of the fragment length distributions from the six cluster fibers showed they were similar at the 95 % confidence level (p = 0.9265). The smaller average fragment lengths of the 1 and 6 fibers (end fibers) reflect an increase from 38 breaks observed in the core fibers (2 through 5) to 39 and 40 breaks, respectively. The p-value for the fragment lengths of the core fibers was found to be 0.9995, while the p-value between the end fibers was 0.7473. The lower correlation values on the edge fibers may reflect edge effects due to the presence of only one adjacent fiber.

3.3 Coordinated fiber breaks at saturation (actual versus theoretical). A plot of the actual fiber break locations in the cluster fibers is shown in Figure 4. These results indicate that 38 of the fiber breaks in each fiber are aligned, with the end fibers containing 1 and 2 additional fiber breaks, respectively. These additional breaks are highlighted by red rectangles in the figure. The average dispersion of the aligned breaks was found to be (72 ± 30) µm, where ± denotes 1 standard deviation. Additional calculations showed that 63 % of the dispersion values are within 1 standard deviation of the mean and 97 % of the values are within 2 standard deviations.

Theoretical break locations based on the uniform distribution assumption are shown in Figure 5. Aligned fractures of all 6 breaks are theoretically predicted on the ends of the analyzed data. The non-alignment in the center is due to the 1 and 6 fiber containing 39 and 40 over the calibrated length rather than the 38 breaks found in the core fibers. Preliminary analyses of the fiber break locations suggests that the theoretical average dispersion of the breaks must be greater than observed experimentally for the theoretical break positions to be considered aligned or coordinated.

4. CONCLUSIONS

Statistical analyses of the break locations of an E-glass 6-fiber multi-fiber array were found to conform, similar to the results obtained on E-glass single fiber composites [13], to a uniform distribution. However, the break density decreased by approximately 20 % along each fiber in the multi-fiber array relative to those of two single fibers that were also embedded in the sample specimen. The results from this 2-D combinatorial microcomposite test specimen confirm earlier results by Li *et al.* [3] and highlight the fact that the van Dyke and Hedgepeth [4] and Zweben [5] shear-lag model, which is often used to model composite failure, predicts results inconsistent with experimental data.

ANOVA analyses of the fragment lengths at saturation from each fiber in the cluster showed that the distributions were identical at the 95 % confidence level with a p-value of 0.9265. Furthermore, the dispersion of the aligned breaks in the cluster were on average 70 μm and appear to be less than those generated by theoretical predictions, when the actual number of fiber breaks in each fiber are used in the analysis. Finally, since the break locations are uniform, any correlation of the fragment lengths at saturation with distributions such as Weibull, shifted-exponential, and lognormal is fortuitous. Since a composite's strength is primarily controlled by the fiber, these results may have only a minor impact on this parameter. However, these results may allow a more accurate prediction of energy absorption and durability of the composite.

5. REFERENCES

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Table 1. Statistic information and fragment lengths of the cluster fibers

Fibers (#)	**1**	**2**	**3**	**4**	**5**	**6**
Average fragment length (µm)	414.913	426.512	428.017	425.359	426.341	407.222
Correlation coefficient	0.9996	0.9995	0.9992	0.9995	0.9995	0.9993

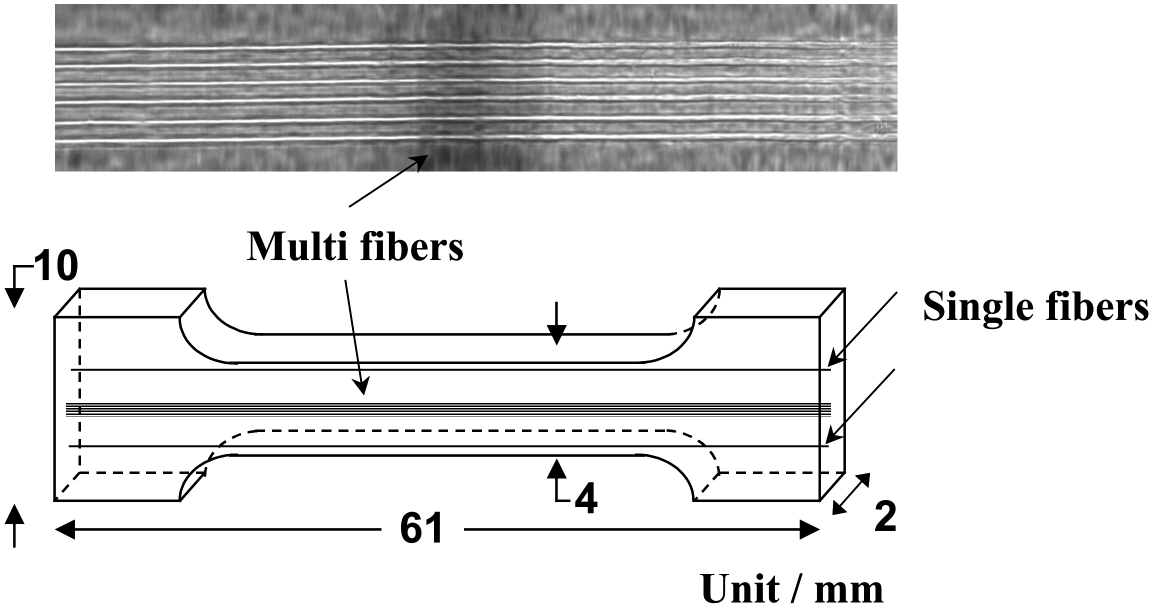

Figure 1. Schematic of a 2-D combinatorial microcomposite containing a 2-D multi-fiber array and two single fibers. A photo of typical 6-fiber array is also shown.

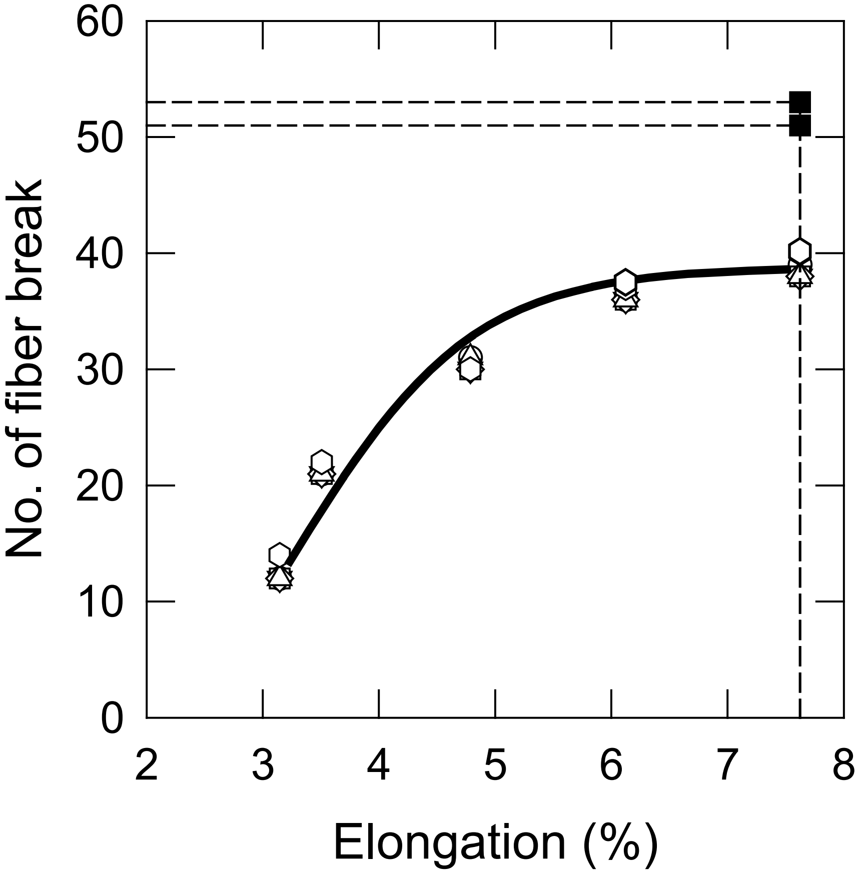

Figure 2. Fiber break evolution in cluster fibers with increasing evolution (open symbols). Solid symbols indicate the number of fiber breaks at the end of the test in the single fibers and line is drawn to aid the reader's eyes

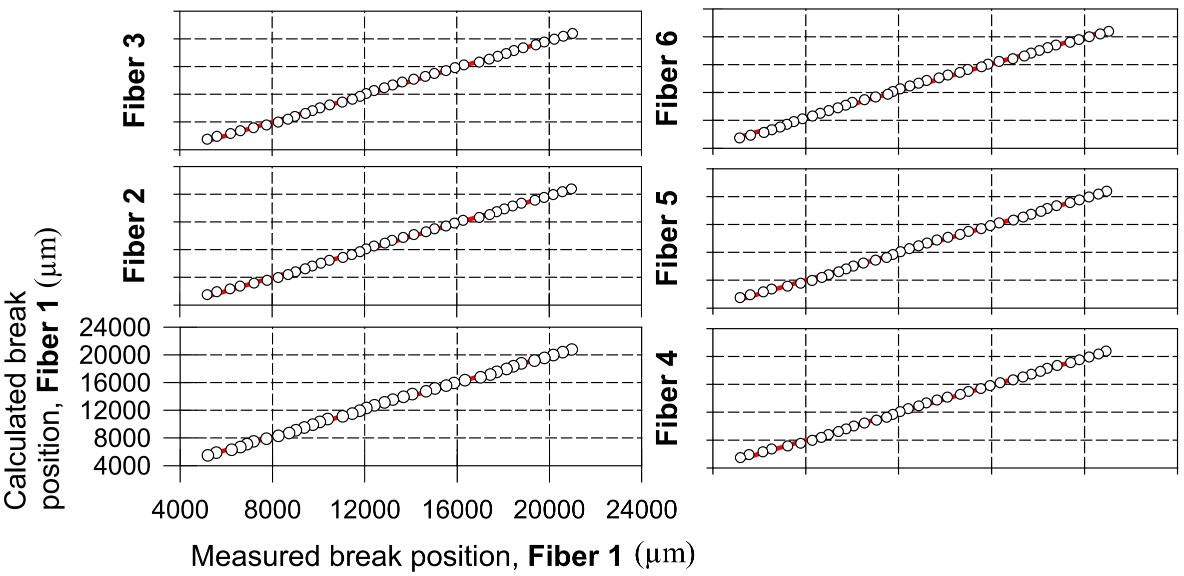

Figure 3. Probability plots of cluster fiber breaks assuming the fiber breaks are uniformly spaced. The solid line represents the empirical fit.

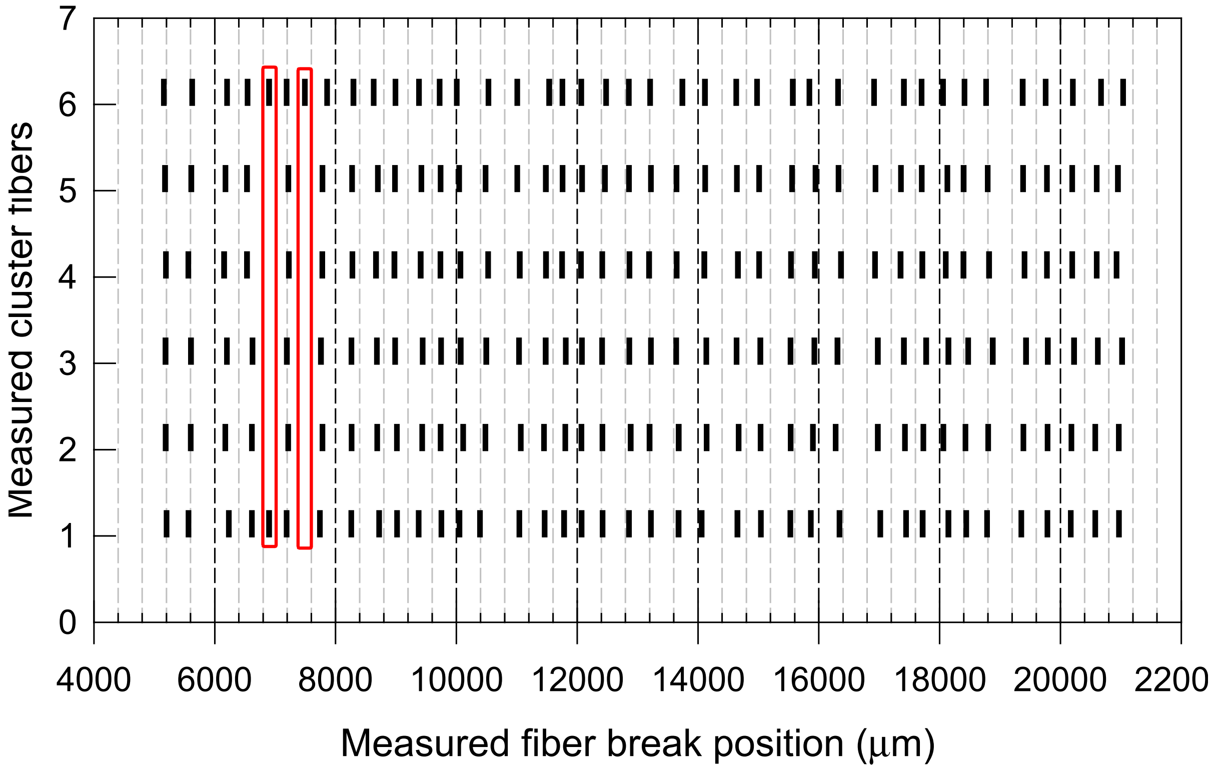

Figure 4. Plot of measured fiber break locations in the multi-fiber array. Vertical lines added to aid visualization of aligned fracture breaks.

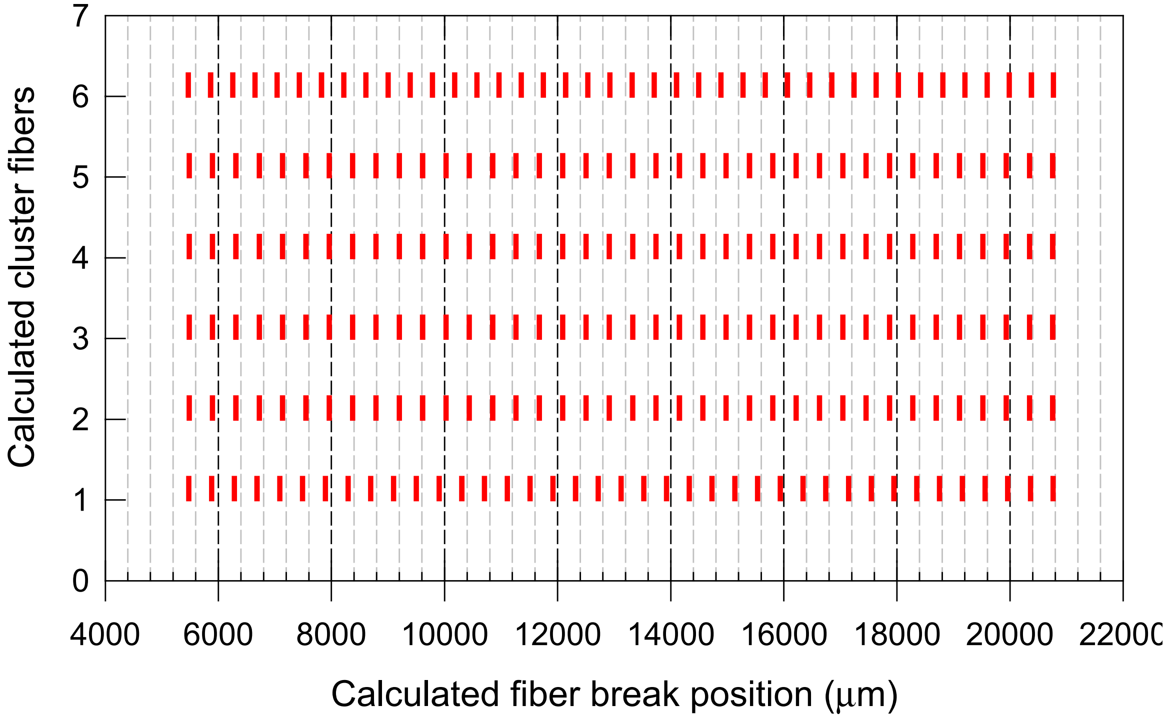

Figure 5. Plot of theoretical fiber break locations in multi-fiber array based on uniform distribution assumption. Vertical lines added to aid visualization of aligned fracture breaks.