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## Determination of Interfacial Shear Strength in Epoxy/Glass Composites by Multi-Fiber Fragmentation Test (MFFT)

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### 1. Introduction

The mechanical strength of a structural composite is strongly affected by the strength and toughness of the interface achieved between the continuous matrix phase, or resin, and the reinforcing phase, normally consisting of multiple arrays of carbon or glass fibers. [1] Interfacial shear strength (IFSS) is the accepted parameter for quantifying the strength of the matrix-fiber interface. However, the value of IFSS is not directly accessible by measurement and must be approximated indirectly from other data.

One mechanical test which generates such data is the single fiber fragmentation test (SFFT). [2-4] In SFFT, a single fiber is embedded in a thermoset resin. Once fabricated, the composite specimen is strained under tensile conditions in timed increments. During this strain process, breaks occur in the fiber to relieve stress on the composite. As the test progresses, more breaks occur in the fiber until a time is reached where no further breaks occur. This point represents the stress saturation of the fiber. Further deformation on the specimen after this point results in no additional breaks. By recording the overall strain and load on the fiber at saturation, as well as the number of breaks and break fragment lengths, an approximate calculation for the interfacial shear strength may be made using an elementary mechanical force balance.

In this paper we extend the SFFT technique to multiple fiber arrays (multi fiber fragmentation test MFFT) to more closely study the effect of fiber-fiber interactions on stress transfer and simplifying assumptions about the matrix. We use MFFT data obtained from a conventional epoxy / E-glass system to show that the spatial distribution of fiber breaks along the axes of fibers in close bundles is

fitted best by a Uniform rather than a Weibull distribution.

A representative statistical distribution of break fragment lengths can improve the accuracy of calculations for local and global IFSS in micromechanical specimens.

### 2. Materials and Methods†

Diglycidyl ether of bisphenol-A (DGEBA, Epon 828, Shell Co.) 1,4 butanediol diglycidyl ether, (DGEBD, RD-2, Ciba-Geigy) and metaphenylene diamine, (m-PDA, Fluka Chemical Co.) were used in the mass fraction ratio 100:25.1:20.6 for the matrix. E-glass fibers treated with 3-amino propyl triethoxysilane (A-1100) were used.

Multi-fiber fragmentation specimens (dogbones) were made using the procedure for a single fiber described by Drzal. [5] The resin mixture was cured for 3 h at 60 °C and 2 h at 125 °C.

These specimens were then mounted in a custom-built Multi-Fiber Fragmentation Tester. [6] Using the automatic loading device, the fragmentation specimen was deformed by sequential increments (step strains) with a loading rate of 0.85 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 were obtained by taking an image of the specimen after each loading step using a digital camera (25x magnification). Break locations in each photograph were digitized manually using the *Digimizer* software.

The break coordinate data were then fitted to a number of statistical distributions to determine their goodness-of-fit. Although the most commonly used distribution is the two-parameter Weibull distribution, we attempted to fit the data with a

Uniform distribution based on previous findings for SFFT. [3] The expected locations of the fiber break centroids may be calculated using appropriate formulas for the Uniform and Weibull distributions. [7, 8]

### 3. Results and Discussion

The first result from the MFFT is the distribution of breaks in the fiber or fibers of interest. This is because break location and break length are key variables in the calculation of interfacial shear strength. For example, the change in break distribution of a single control fiber subjected to ten minute strain increments over 360 min is shown in Figure 1. The sorted break locations (y axis) are plotted against the percentiles of a uniform distribution (x axis). It can be seen that there is a tighter adherence of all points to the 45° test line with test time, and a progressively increasing value for the Pearson probability correlation coefficient, (PPCC), from 0.9993 at 200 min to 0.9996 at 360 min, demonstrating the uniformity of the spatial distribution of breaks on the single fiber. A satisfactory PPCC value is considered to be greater than 0.99.

Corresponding graphs for the individual fibers of a six-fiber bundle are shown in Figures 2 to 7. Once more, it can be seen that for each of these fibers, there is a tighter adherence of all points to a 45° test line with test time, and a progressively increasing value for the PPCC.

### 4. Conclusion

A multi-fiber fragmentation test has been applied to parallel arrays of E-glass fibers under uniaxial tension embedded in a DGEBA matrix. The adherence of the distribution to uniform behavior is measured using PPCC that are higher than those obtained when using either two- or three- parameter Weibull models to describe the same distribution. This represents a significant departure from previous descriptions of such multi-fiber systems in the literature. The PPCC of the probability plots with uniform distribution was significantly high ranging from 0.9993 to 0.9996, and this has revealed an underlying uniform spatial distribution of fiber breaks in each individual fiber. Further analyses are being performed to more precisely compare the use

of the Uniform distribution with the two-parameter Weibull distribution in MFFT systems.

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### †Note

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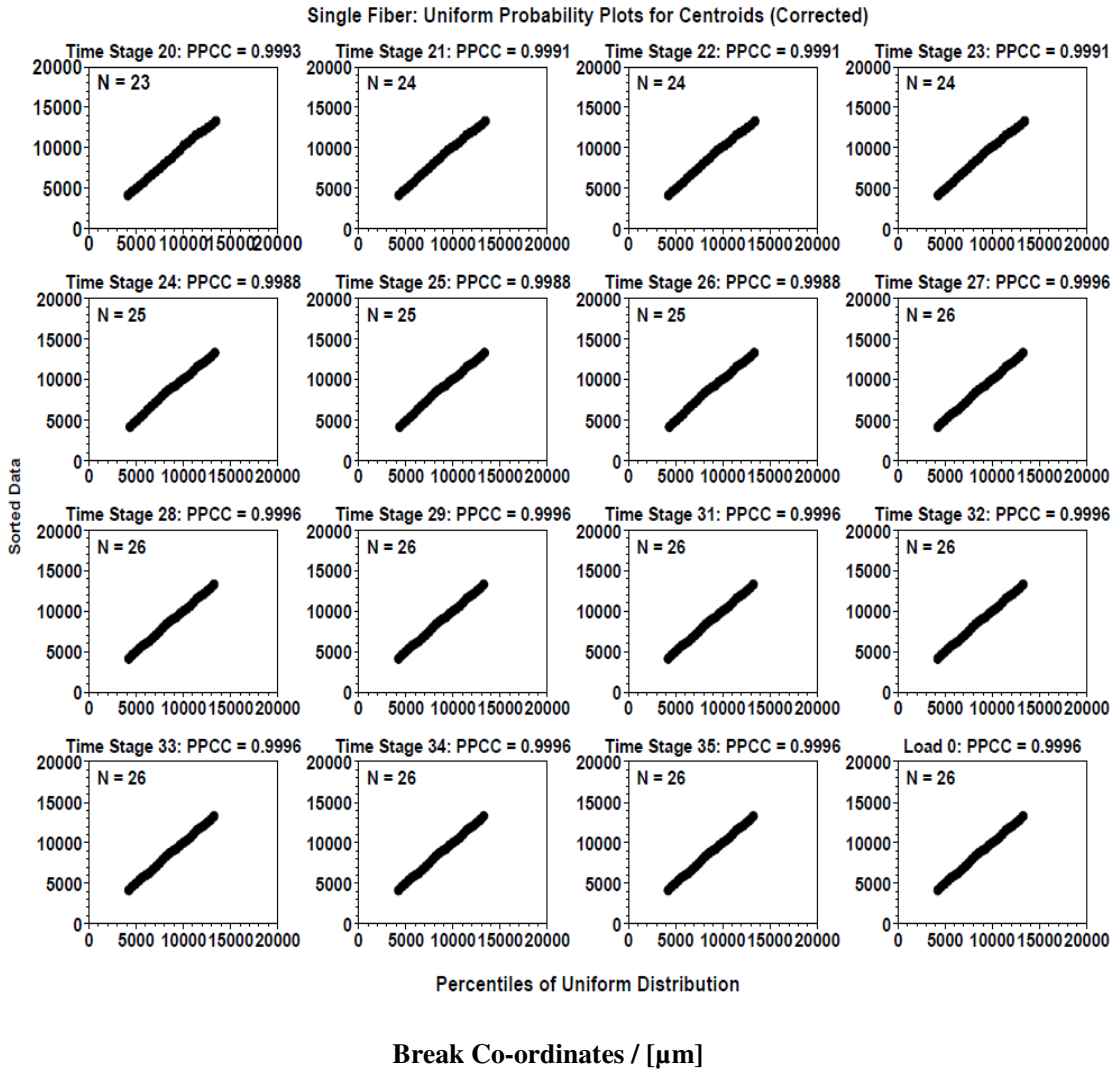
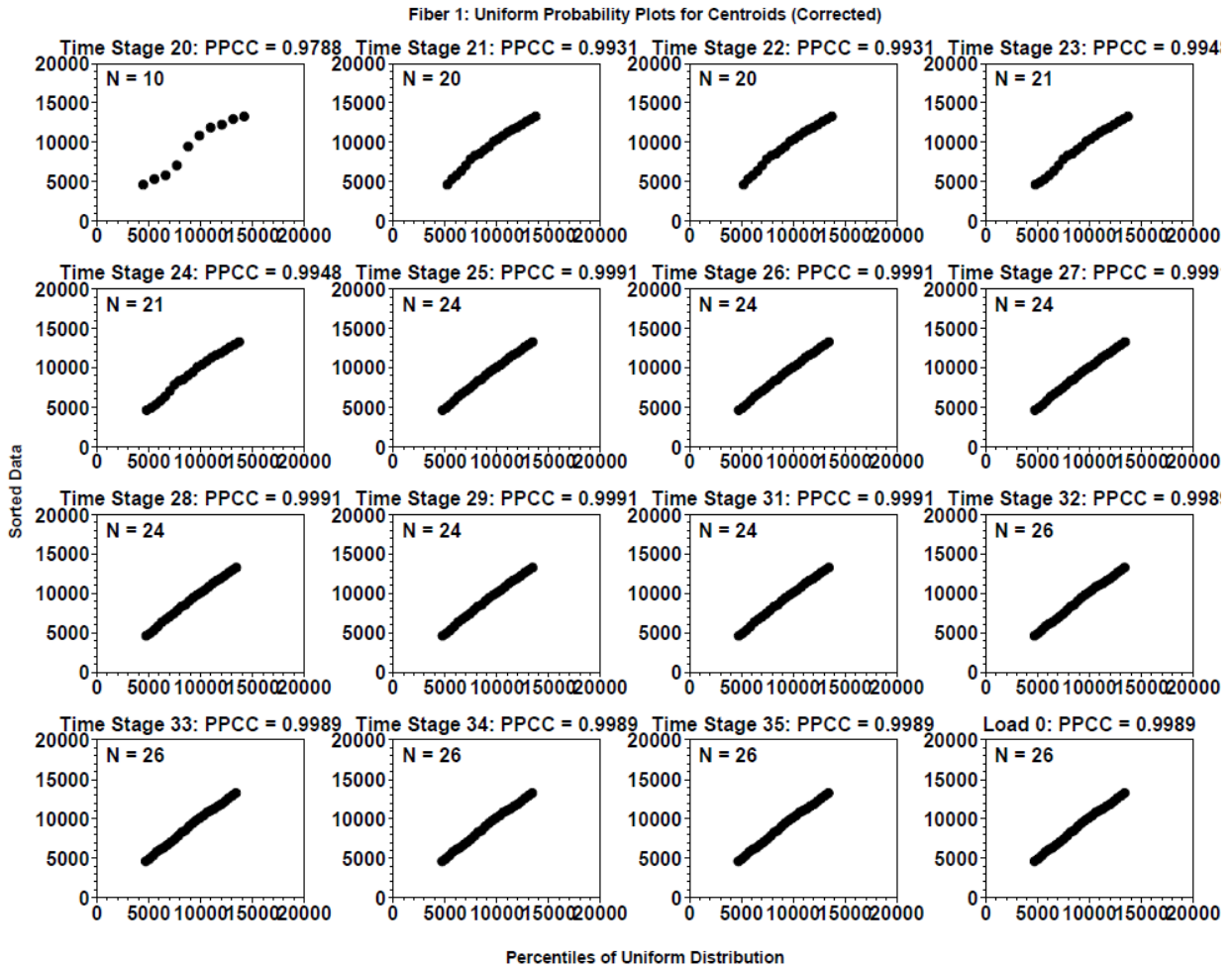
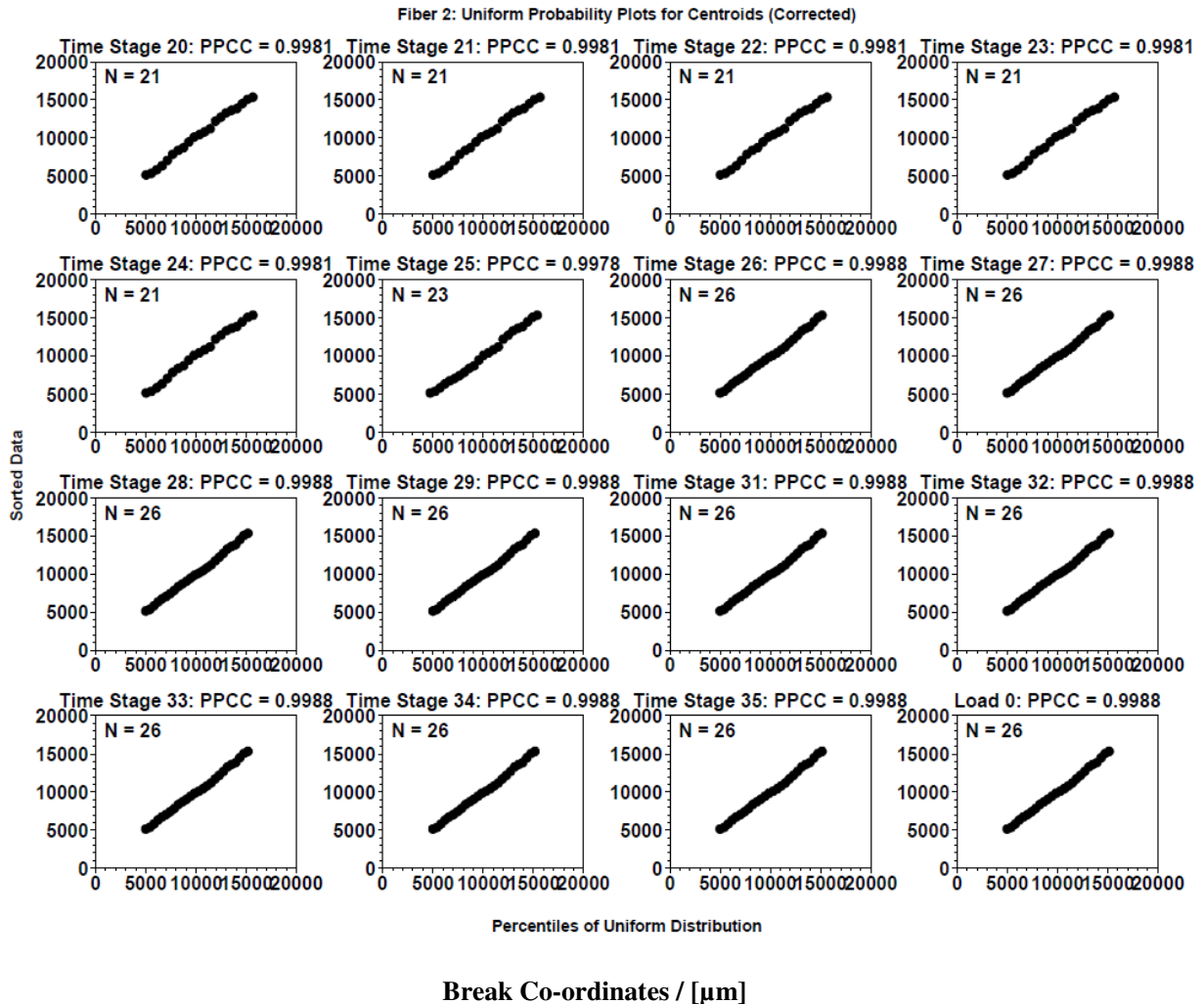


Figure 1 Uniform Plot for break locations of a single fiber every ten minutes for 360 minutes. Actual break co-ordinates (Y-axis) are plotted against co-ordinates calculated for a Uniform break centroid distribution (X-axis). Final plot (bottom right, Load 0) plots co-ordinates for Stage 35 when specimen has been relaxed from its final deformation.

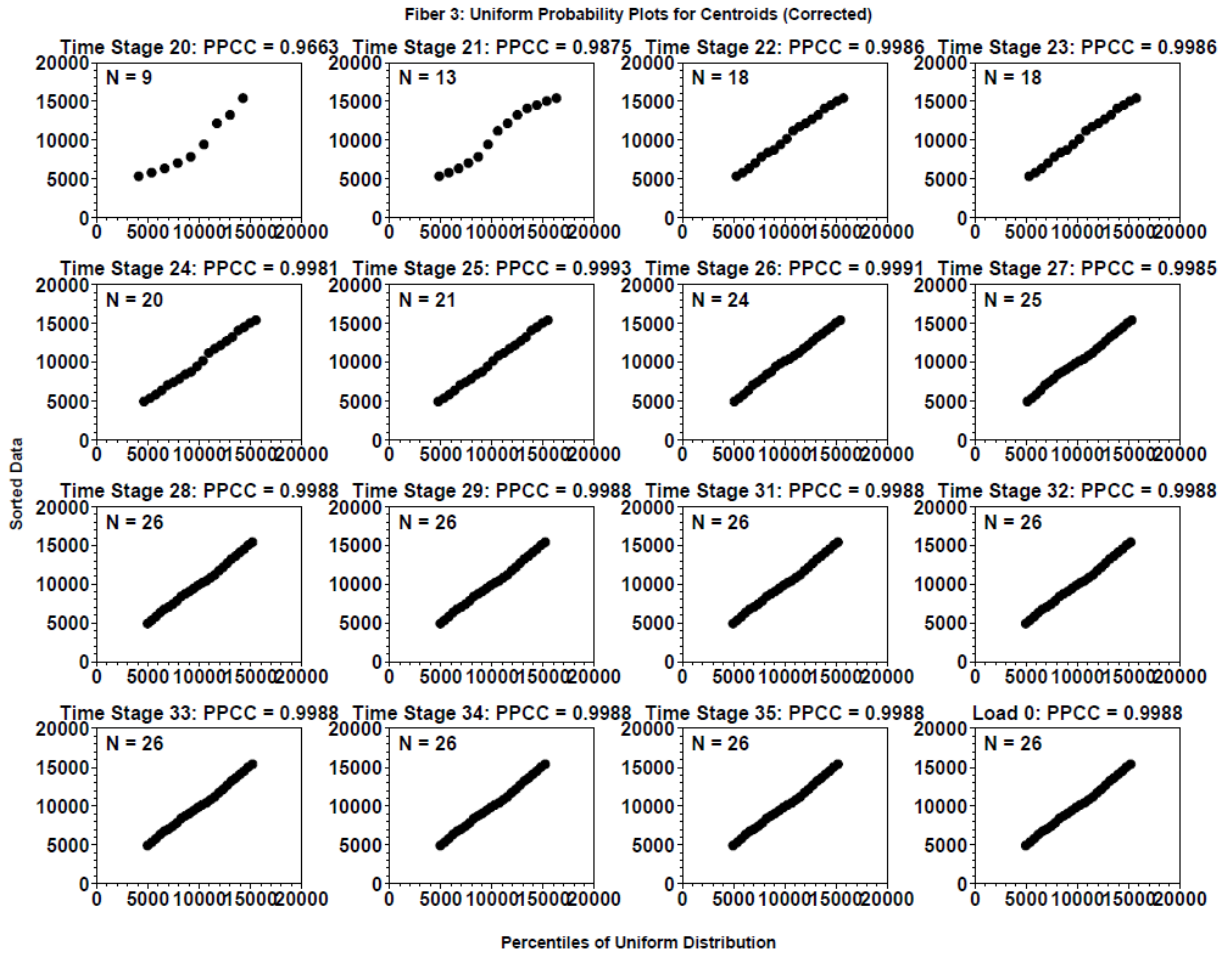


**Figure 2** Uniform Plot for break locations of Fiber 1 in a six fiber bundle every ten minutes for 360 minutes. Actual break co-ordinates (Y-axis) are plotted against co-ordinates calculated for a Uniform break centroid distribution (X-axis). Final plot (bottom right, Load 0) plots co-ordinates for Stage 35 when specimen has been relaxed from its final deformation.

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**Figure 3** Uniform Plot for break locations of Fiber 2 in a six fiber bundle every ten minutes for 360 minutes. Actual break co-ordinates (Y-axis) are plotted against co-ordinates calculated for a Uniform break centroid distribution (X-axis). Final plot (bottom right, Load 0) plots co-ordinates for Stage 35 when specimen has been relaxed from its final deformation.



Break Co-ordinates / [ $\mu\text{m}$ ]

Figure 4 Uniform Plot for break locations of Fiber 3 in a six fiber bundle every ten minutes for 360 minutes. Actual break co-ordinates (Y-axis) are plotted against co-ordinates calculated for a Uniform break centroid distribution (X-axis). Final plot (bottom right, Load 0) plots co-ordinates for Stage 35 when specimen has been relaxed from its final deformation.

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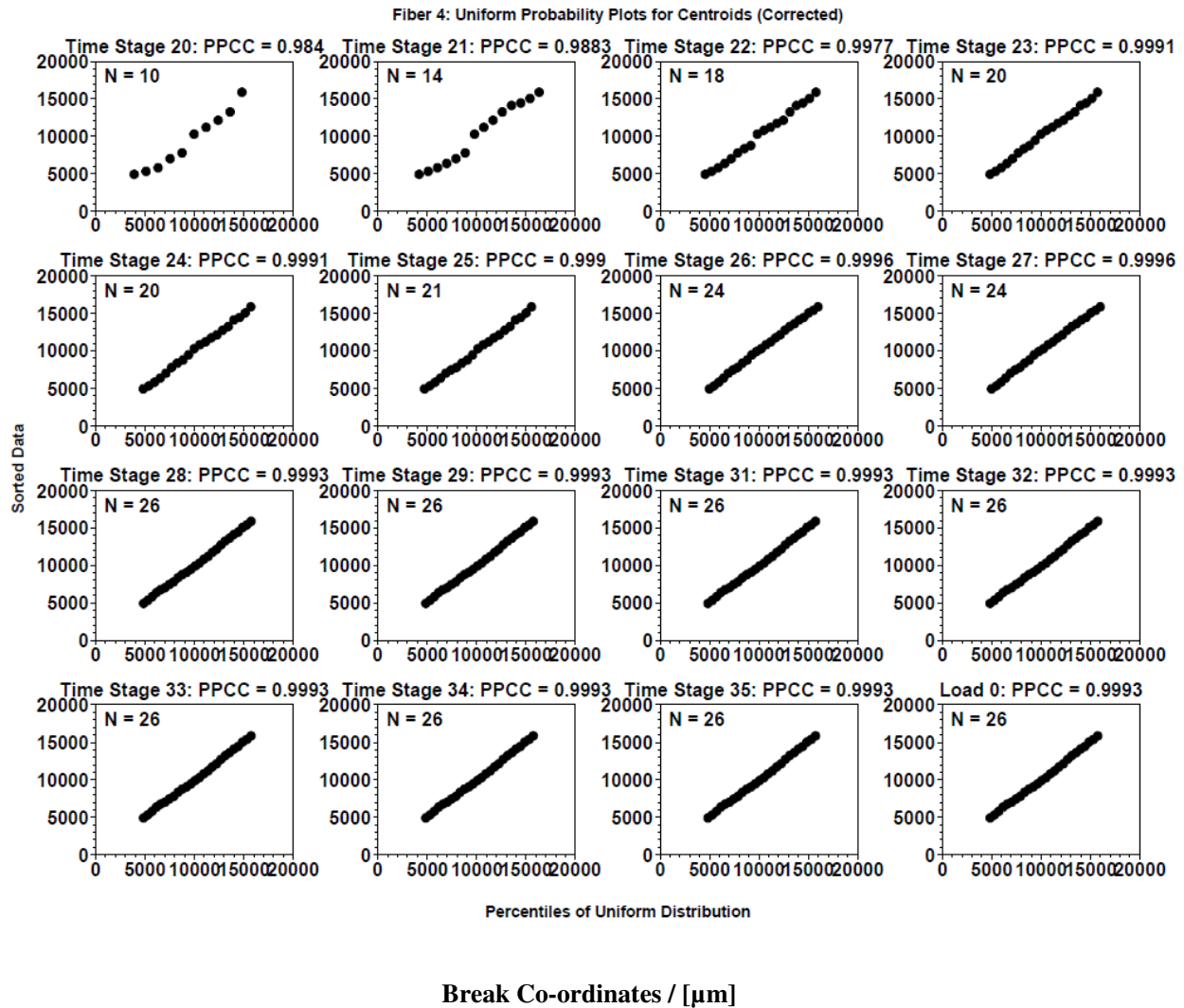
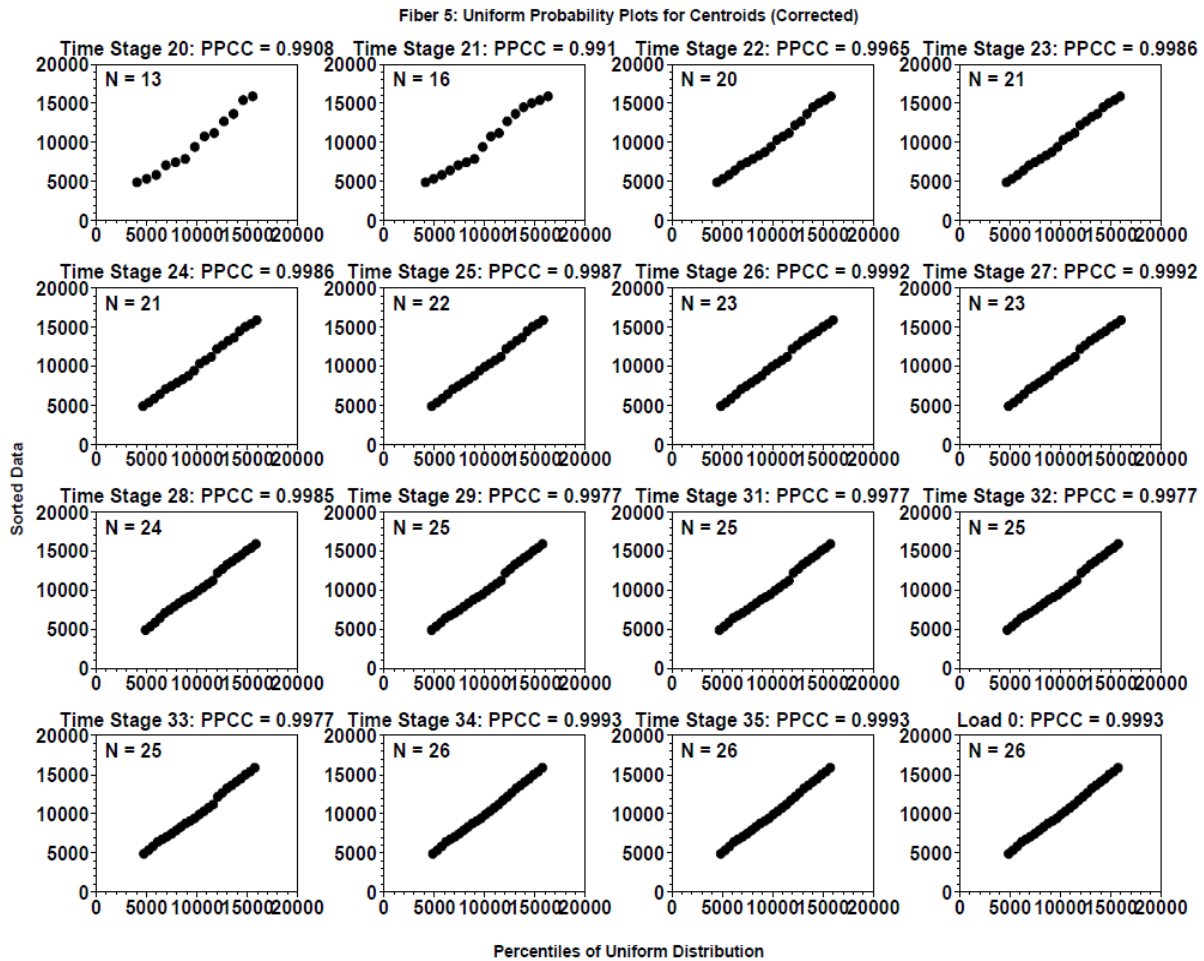


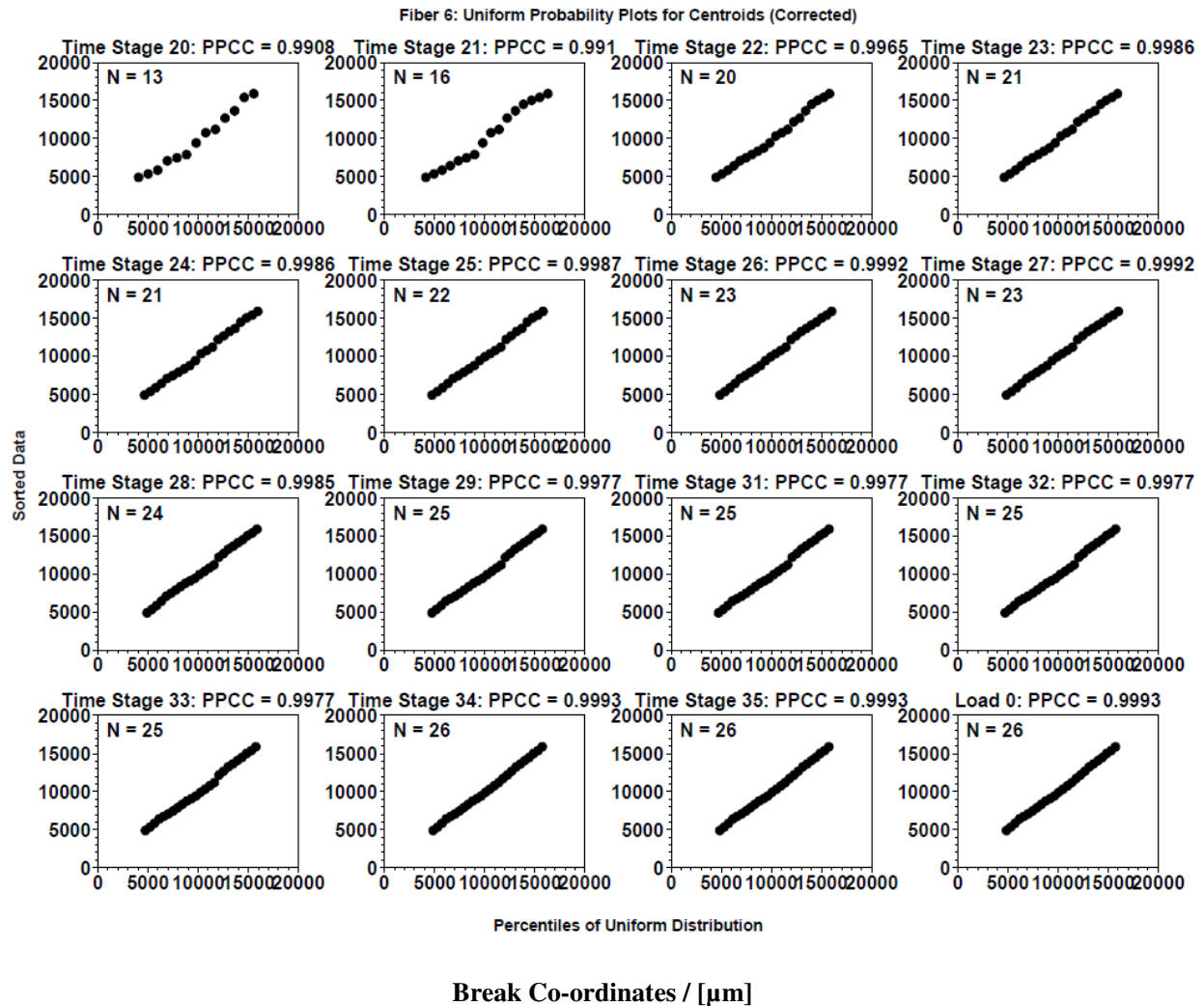
Figure 5 Uniform Plot for break locations of Fiber 4 in a six fiber bundle every ten minutes for 360 minutes. Actual break co-ordinates (Y-axis) are plotted against co-ordinates calculated for a Uniform break centroid distribution (X-axis). Final plot (bottom right, Load 0) plots co-ordinates for Stage 35 when specimen has been relaxed from its final deformation.





**Figure 6** Uniform Plot for break locations of Fiber 5 in a six fiber bundle every ten minutes for 360 minutes. Actual break co-ordinates (Y-axis) are plotted against co-ordinates calculated for a Uniform break centroid distribution (X-axis). Final plot (bottom right, Load 0) plots co-ordinates for Stage 35 when specimen has been relaxed from its final deformation.

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**Figure 7** Uniform Plot for break locations of Fiber 6 in a six fiber bundle every ten minutes for 360 minutes. Actual break co-ordinates (Y-axis) are plotted against co-ordinates calculated for a Uniform break centroid distribution (X-axis). Final plot (bottom right, Load 0) plots co-ordinates for Stage 35 when specimen has been relaxed from its final deformation.