STRENGTH AND STRUCTURE OF COTTON FIBER DURING DEVELOPMENT You-Lo Hsieh University of California, Davis

<u>Abstract</u>

Single fiber strengths and crystalline structure of greenhouse-grown Maxxa Acala cotton fibers at varying stages of development from 14 days pass anthesis to maturity are reported and compared with those of SJ-2 cotton fibers. The forces required to break single fibers increase most significantly during the fourth week and are similar between SJ-2 and Maxxa varieties through the end of the fourth weeks of development. Beyond 30 dpa, both the single fiber breaking forces and tenacities of the Maxxa cotton fibers are higher than those of the SJ-2 cotton fibers. For both varieties, cellulose I crystalline structure is found in fibers at all stages of development and the crystallinity

and apparent crystallite sizes in the 101, $10\overline{\mathbf{I}}$ and 002 plane directions increase with fiber development. Maxxa fibers have larger crystal sizes but lower crystallinity and lower crystal density than the SJ-2 fibers at the same age. Within each variety, the single fiber breaking forces increase with increasing overall crystallinity and crystallite sizes. The relationships between the single fiber breaking tenacities and crystallinity are, however, different between these two varieties.

The relationships between cotton fiber structure and strength have been the subjects of studies. The strength of cotton fibers is attributed to the rigidity and the high molecular weight of the cellulose chains, the extensive intermolecular and intramolecular hydrogen-bonding, and the highly fibrillar and crystalline structure of the fibers. Cotton fiber strength has been shown to be associated with molecular weight of the cellulose, crystallinity, and reversal and convolution characteristics of the fibers. These relationships have, however, not been clearly verified on developing cotton fibers.

This paper reports the single fiber breaking strength of developing cotton fibers and the strength-structure relationship. Two Acala varieties, i.e., Maxxa and SJ-2, were included. The tensile measurements were performed using Instron tensile tester as well as the Mantis single fiber instrument.

Experimental

Fibers Bolls from twenty-two Maxxa plants and nine SJ-2 plants grown under the greenhouse conditions were used for this study. Flowers were tagged on the day of flowering (anthesis). The first position (closest to the main stem)

bolls were randomly sampled between the fourth and the twelfth fruiting branches. This boll-sampling method has been confirmed not to cause variation among single fiber strength during development. Previously established procedures for boll storage and fiber preparation for single fiber strength measurements were followed [1].

Single Fiber Strength and Property Fiber from five most developed ovules from each boll were used for tensile All single fiber measurements were measurements. performed on the middle portion of fibers taken from the middle sections of the ovules or seeds. Tensile measurements of developing SJ-2 and Maxxa fibers were performed at a 50-mm/min strain rate using an Instron tensile tester (1122 TM) equipped with standard pneumatic and rubber-faced grips. For Maxxa fibers, tensile measurements were also performed using the Mantis single fiber instrument. All tensile measurements were performed with a 3.2 mm-gauge length and under a constant temperature of 21°C and a 65% relative humidity. The linear densities or tex (grams per kilometer length) of fibers were measured using fibers from mid-ovule areas of the same seeds. The middle one centimeter sections of fibers were cut from an array of combed and aligned fibers. Onehundred 1-cm fibers were weighed to 0.1 mg and five such measurements were made from each boll. The means and standard deviations of the linear densities are reported.

Wide Angle X-ray Diffraction Wide angle X-ray diffraction (WAXD) of fibers ground in a Wiley mill to pass through a 20-mesh screen was performed using a Scintag XDS 2000 X-ray diffractometer. Diffraction intensities were counted at 0.05°-steps between a 5° to 40° two theta (2q) angle range. The WAXD scattering was corrected, normalized and resolved into the non-crystalline scatter background and peaks. A mixture of Gaussian and Cauchy functions were employed to achieve best curvefitting. Crystallinity is calculated as the ratio of the summation of all resolved peaks to the total scatter under the unresolved trace including the background. The apparent crystallite sizes of the $101, 10\overline{1}$ and 002 reflections planes is based on the Sherrer equation. Further details on the experimental procedures and spectra analyses can be found in our previous paper [3].

Results and Discussion

Both the breaking forces measured by the Mantis instrument show that the hydrated and dried single Maxxa fibers increase with fiber development (Figure 1a). Hydrated fibers have higher breaking forces than the dried fibers whereas the breaking elongation values are higher for the dried fibers (Figure 1b). The breaking forces of the dried fibers increase more between 20 and 30 dpa than in the later stages. The work to break values of both the hydrated and dried fibers are similar (Figure 1c). The breaking forces measured by the Mantis instrument appear to be slightly higher than those by the Instron (Figure 2a) whereas the opposite is observed on the breaking elongation(Figure 2b).

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Single fiber breaking forces are similar between SJ-2 and Maxxa varieties during the first 4 weeks of development. Between 30 and 50 dpa, both the forces required to break single fibers and the breaking tenacities of the Maxxa fibers are higher than the SJ-2 fibers. Although the single fiber breaking tenacities of the SJ-2 cotton fibers do not appear to vary at the beyond 21 dpa, the breaking tenacities of the Maxxa cotton fibers continue to increase with the secondary cell wall thickening process. Because of the small numbers of samples involved here, the relationship between tenacity and fiber development requires further investigation.

WAXD of the developing SJ-2 and Maxxa fibers shows four peaks located near 2θ angles of 14.7°, 16.6°, 22.7° and 34.4° , characteristic of the 101, $10\overline{1}$, 002 and 040 reflections of cellulose I, respectively. The overall crystallinity and apparent crystallite sizes of fibers from both varieties increase with fiber development. The unit cell sizes slightly decrease and thus the crystal densities increase with fiber Among the crystal lattice planes, the development. alignment of the glucosidic rings in respect to the 002 planes improves most significantly with fiber cell development. The crystallinity and crystal density of SJ-2 fibers are higher than those of Maxxa fibers during the fifth and sixth weeks of fiber development. The 002 and 101 crystallite dimensions of Maxxa fibers, on the other hand, are larger than those of the SJ-2 fibers. These increases coincide with the largest increase in forces to break single fibers.

Within each variety, positive relationships are observed between single fiber breaking forces and the overall crystallinity as well as between single fiber breaking force and crystallite size. However, the relationships between tenacities and crystallinity are different between these two varieties. The increased crystallinity and crystallite sizes and perfection offer only partial explanation to the strength development of cotton fibers. Further investigation on the varietal differences and tenacity-structure relationships is underway.

Summary

- For both SJ-2 and Maxxa varieties, the single fiber breaking forces increase most significantly during the fourth week of fiber development.
- The forces required to break single fibers are similar between SJ-2 and Maxxa varieties through the end of the fourth week of development. Beyond 30 dpa, both the single fiber breaking forces and tenacities of the Maxxa cotton fibers are higher than those of the SJ-2 cotton fibers.
- The linear density and seed fiber weight of the Maxxa fibers are lower than those of the SJ-2 fiber when compared at the same developmental stages.
- The cellulose **I** crystalline structure is clearly evident at the early developmental stage of 21 days post anthesis (dpa) and remains unchanged throughout fiber

development. The overall crystallinity and the apparent crystallite sizes increase with fiber development for both varieties.

• At the same developmental stages, Maxxa fibers have larger crystal sizes but lower crystallinity and lower crystal density than the SJ-2 fibers. Within each variety, the single fiber breaking forces are positively related to both the overall crystallinity and crystallite sizes.

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Figure 2. Single fiber breaking properties of Maxxa cotton measured by the Instron and Mantis instruments.

Figure 1. Single fiber breaking properties of Maxxa cotton measured by the Mantis instrument.