The characterization of glass fibers for papermaking

R. M. Bailey and W. T. McKean

Research associate and professor chemical engineering and pulp and paper science, Dept. of Pulp and Paper Science, College of Forest Resources, University of Washington, Seattle, Wash. 98195

ABSTRACT *By examining both physical and surface properties, we characterized glass wool fibers used for papermaking. Going beyond normal measurements of mean values, we examined the individual fiber properties of length, diameter, distribution, and electrophoretic mobility. We also examined the fiber interactions that impact sheet formation and strength. Sheets made from long or thin fibers gave higher tensile strength than those made from short or wide fibers. Direct measurements by microprojection methods revealed wide distributions of length and diameter. In direct measurements of diameter, we determined differences previously undetected by the methods that measure only average values. We also measured surface charge to help predict fiber interactions prior to sheet formation.*

KEYWORDS
Glass fiber
Formation
Nonwovens
Papermaking
Surface charge

Glass fibers used in the nonwovens, composite, and specialty paper industry are normally produced in the form of either chopped strand or wool. Wool grades are generally smaller in diameter and have wider ranges of distribution in length and diameter than the grades made of chopped strand. As commonly known, fiber dimensions influence paper properties. Despite the dominance of short fibers in the headbox furnish, wool grades may be used, nevertheless, to produce surprisingly strong paper.

On the other hand, the broader distributions for wool grades make it more difficult to determine the length and diameter dimensions. Adequate statistical measurements of the random wool properties require tedious repetitions that work against a thorough characterization of the wool fiber properties. Common methods to relate wool fiber properties to papermaking properties include: air resistance of fiber pads, settling tests, and pad compressibility. These methods are useful for quality control in fiber manufacturing and can be related to paper tensile tests (*1*, *2*). However, they provide only a mean value for length and diameter, with no measure of the distribution of these quantities. Some results suggest that changes in the length and diameter distribution that are not detected by these tests may lead to differences in paper properties (*3*).

Furthermore, little attention has been given to how fiber surface properties, such as surface charges, influence the strength of binderless and bonded glass papers. The surface property most commonly measured in the paper industry is the surface charge, also referred to as "zeta potential" or "electrophoretic mobility." Surface charges are known to cause an impact on fiber interactions, thus influencing sheet formation and strength (*4*). Consequently, in our study, we focused on measuring physical dimensions, surface properties, and related paper properties for some glass wool fiber grades with mean diameters of about 0.8–7.3 μm.

Experimental methods

Here, we will describe briefly the methods we used to directly measure fiber diameter and length distributions. The Kolmogorov-Smirnov test was used to compare these distributions with log normal distributions and with other fiber samples (*5*). Complete descriptions of these procedures and measurements of fiber surface charge are available elsewhere (*6*).

Fiber length

In making direct measurements of fiber length distributions, we deposited dispersed glass fibers on microscope slides. The images from these slides were projected through a light microscope onto the tablet of a Zeiss digitizer, Model 100, at 35–75X. The fiber lengths were measured manually with the aid of the digitizer. Between 250 and 500 fibers were measured for each sample.

Fiber diameter

Photographs of the scanning electron

microscope image taken at 8000X were enlarged to achieve a total magnification of 17,884X. The scanning electron microscope was a Joel JSM-840 A. The Zeiss digitizer was used to make direct measurements of fiber diameters from the enlarged photographs. Fifty fibers from each sample were measured.

Fiber surface electrical charge

The zeta potential was measured using a Laser Zee model 500 zeta meter, manufactured by Pen Kem, Inc. This particular model measures the electrophoretic mobility and automatically converts it to zeta potential using the Smoluchowski equation. The unit is calibrated for aqueous solutions at 20°C. Mobility, in units of m^2/Vs, may be calculated by dividing the zeta potential readout, in units of mV, by 14.2×10^8 (*7*).

The charge at the surface of the fibers was changed by adjusting the pH of the slurry with sulfuric acid or sodium hydroxide. The samples were held for 30 min before the final pH and zeta potential were measured. Fifty fibers from each sample were measured.

Hydropulping

We used the British Disintegrator, a Waring Commercial Blender, and a 100-L Black Clawson pilot-scale hydropulper to disperse fiber slurries at pH 3 and 0.58% consistency.

Tensile testing

Handsheets were made according to a modified TAPPI Test Method T 205. These were weighed, and the tensile strength was tested according to TAPPI Test Method T 404 on an Instron, Model TML (*6*).

Results and discussion

Fiber length

With few exceptions, paper tensile strength is proportional to fiber length, and paper made from glass wool fibers follows this tendency (*8*). While manufactured wool fibers usually exceed 10 mm, processing in papermaking equipment causes a dramatic drop in the mean length. The mean fiber length and the length distribution are highly influenced by the fiber diameter, stiffness, and type of processing equipment and conditions.

1. Sequence of fiber dispersion showing (A) glass wool as received from the manufacturer, (B) semi-dispersed glass wool, and (C) dispersed glass wool

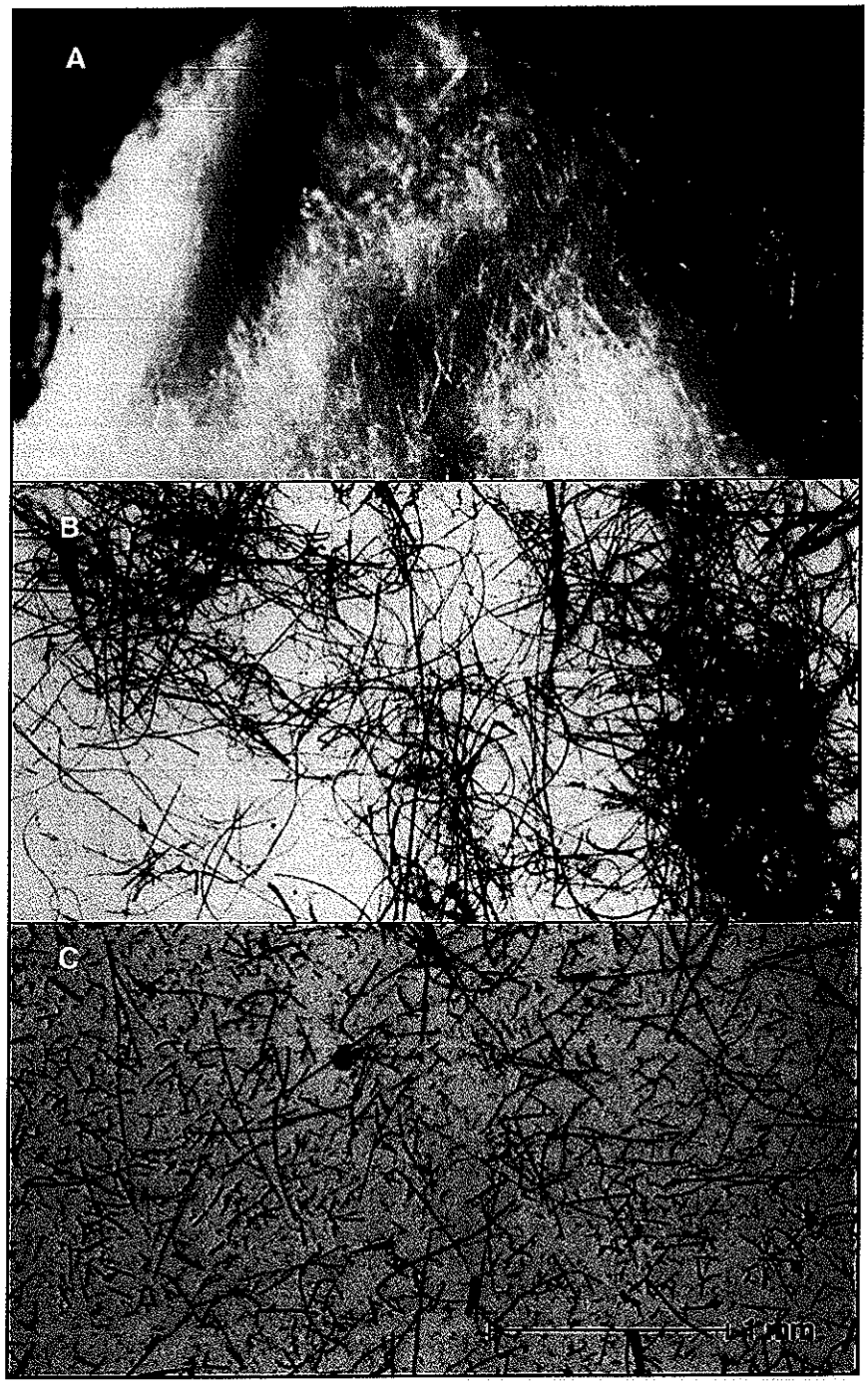
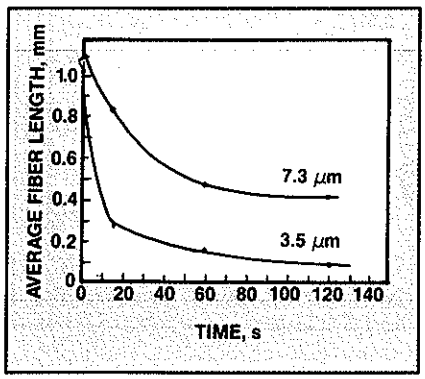


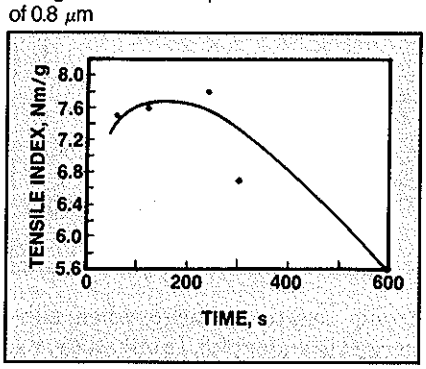
Figure 1 illustrates the typical changes in fiber length that occur during hydropulping. In the manufactured state, as shown in Fig. 1A, wool fibers are long and highly entangled. To form a uniform sheet, these fibers must be dispersed by hydropulping or some other type of high shear mixing. During dispersing, the brittle glass wool fibers break into shorter segments as they are pulled from the entangled fiber bundles. At intermediate hydropulping times, as shown in Fig. 1B, these fiber fragments are distributed between bundles that continue to grow smaller. Smooth, well-formed papers can only be manufactured after further hydropulping to the state shown in Fig. 1C. For a fiber grade with a mean diameter of about 0.8 μm, the headbox mean length has typically been reduced to about 0.3 mm.

The change in fiber length with

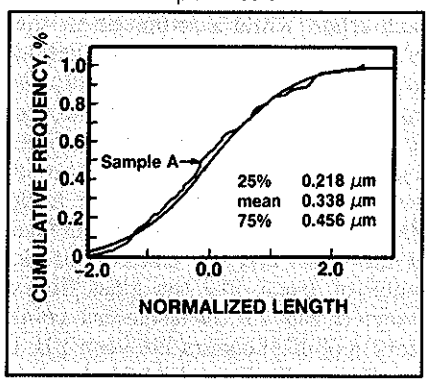
2. The effect of hydropulping time on fiber length, for fibers of two different diameters



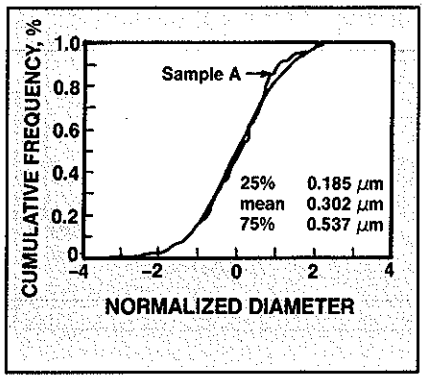
3. The effect of hydropulping time on tensile strength for a fiber sample with a mean diameter of 0.8 μm



4. A comparison of a normal distribution with the normalized natural log of the length distribution of Sample A fibers



5. A comparison of a normal distribution with the normalized natural log of the diameter distribution of Sample A fibers

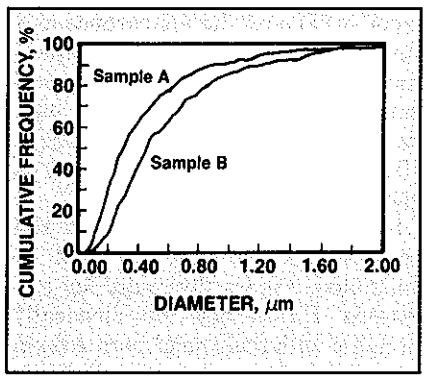


I. Fiber diameter measurements, μm

Sample	Air Frazier[a]	BET[a]	Direct
A	0.64[b]	0.65[b]	0.303
B	0.64[b]	0.70[b]	0.440

[a]Measured by courtesy of Evanite Glass Fiber, Inc.
[b]A standard deviation of 0.02.

6. Comparison of the diameter distribution of Samples A and B



hydropulping time is shown in **Fig. 2** for two grades or diameters of wool fibers. Other fiber grades fall on this plot at positions proportional to their mean diameters. The initial sharp drop in mean length from about 10 mm, reflects the breakage of individual long fibers as they separate from the entangled bundles. Continued hydropulping beyond the state represented in Fig. 1C causes only minor changes in fiber length.

As shown in Fig. 2 and in earlier work on wool fibers, fibers of larger diameter produce longer fibers after hydropulping under the same conditions (*2*). These differences are the result of the relationship between the diameter and fiber strength. Since strength is expressed as force per unit area, it follows that a fiber of larger diameter, which has a larger cross-sectional area, can sustain a larger force than a fiber of smaller diameter. Therefore, thick fibers tend to be stronger and longer than thin fibers pulped for the same amount of time.

As shown in **Fig. 3**, binderless paper tensile strength can be related to the changes in wool fibers that occur during hydropulping. For example, as the original entangled clumps become dispersed and shortened in the early stages of hydropulping, paper formation and tensile strength improve. The optimum hydropulping time varies with fiber grade and process equipment. Hydropulping beyond the optimum duration causes a gradual drop in mean fiber length and tensile strength.

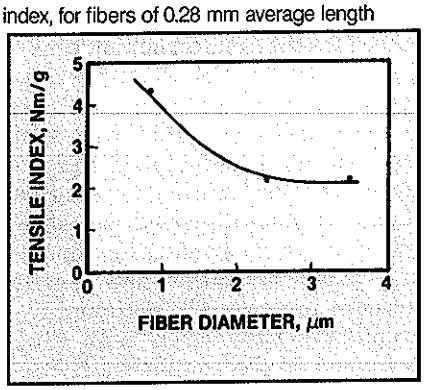
Fracturing of the brittle wool fibers during hydropulping results in a wide distribution of fiber lengths. After hydropulping, for example, a wool grade with a nominal mean diameter of 0.8 μm (which we will designate Sample A) and a mean length of 0.3 mm has a log normal length distribution as shown in **Fig. 4**. Approximately 25% of the fibers are less than 0.1 mm, and 10% are greater than 1.0 mm. Fiber strength and resistance to fracture during hydropulping depends on the diameter. So, for a given grade of wool fiber with a broad distribution in diameter, the thicker fibers will resist breakage more than the finer fibers. Consequently, a broad distribution in diameter contributes to the broad distribution in length.

Fiber diameter

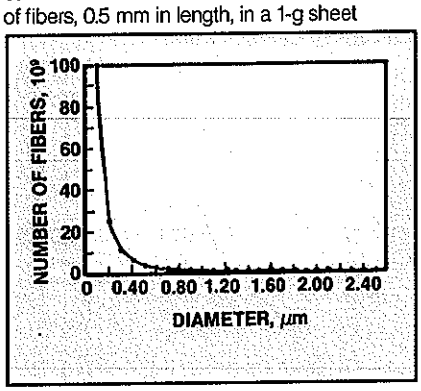
Wool fiber manufacturing processes by their nature produce random distributions in diameter. The various wool fiber grades are labeled on the basis of conventional quality control tests that measure mean diameters. An example of the broad diameter distribution is shown in **Fig. 5**, as log normal for a 0.8-μm-grade fiber. The largest fibers are about 15 times the diameter of the smallest fibers. The diameters of wool fiber grades typically exhibit this type of distribution. Considering the relationship of diameter with cross sectional area, it is not surprising that papermaking slurries derived from these wool fibers also exhibit broad distributions in length.

Different manufacturing processes may produce products with different diameter distributions even though quality control tests yield similar mean diameters. For example, Sam-

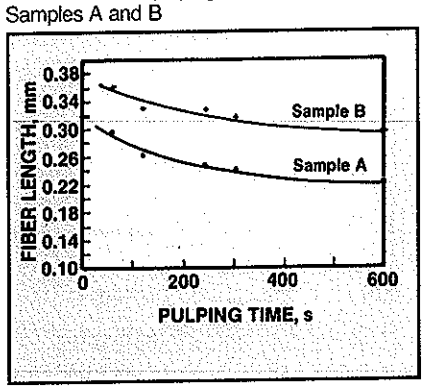
7. The effect of fiber diameter on sheet tensile index, for fibers of 0.28 mm average length



8. The effect of fiber diameter on the number of fibers, 0.5 mm in length, in a 1-g sheet



9. The effect of pulping time on fiber length for Samples A and B



ples A and B in **Table I** are produced by two different manufacturers. These fibers have similar mean diameters as determined by Frazier Air Resistance and Bruneaur, Emmett, and Teller (BET) surface area analysis. Diameter distributions determined from direct measurement and shown in **Fig. 6** indicate that Sample A has a higher fraction of fine fibers than Sample B.

It is commonly known that large-diameter fibers produce substantially weaker sheets than fibers with smaller diameters, for fibers of the same length. The tensile strength of handsheets formed from wool fibers shown in **Fig. 7** confirms this trend. At larger diameters, the tensile strength decreases because the number of fibers per gram of sheet decreases, as shown in **Fig. 8**. Having fewer fibers in a sheet results in fewer fiber contacts per unit length of fiber, and the sheet is weaker. The shape of the curves in Figs. 7 and 8 indicate that the strength of paper made from large-diameter wool grades will be insensitive to upsets in glass fiber manufacturing that may cause changes in mean diameter and diameter distribution. However, in manufacturing fine fiber wool grades (< 1 μm), minor changes in mean diameter and diameter distributions may have considerable impact on the number of fibers per unit mass and on the resulting paper tensile strength.

Quality control tests that result in mean values are useful in detecting and controlling trends in fiber diameter. However, the small differences between Samples A and B may be difficult to detect. Will such differences have an impact on paper properties?

Fiber diameter and fiber length interact to influence paper properties. Thick fibers resist breakage during hydropulping more than thin fibers. So a fiber grade with more thick fibers will tend to have fewer but longer fibers per unit basis weight. In **Fig. 9**, the fiber length of Sample B shows consistently longer mean lengths than Sample A, and Sample B has more thick fibers. Furthermore, a comparison of sheet tensile strengths in **Fig. 10** suggests that Sample B fiber will produce a stronger sheet than Sample A fiber. This indicates that, for these fiber samples, the additional fiber length outweighs the disadvantage of the fiber thickness.

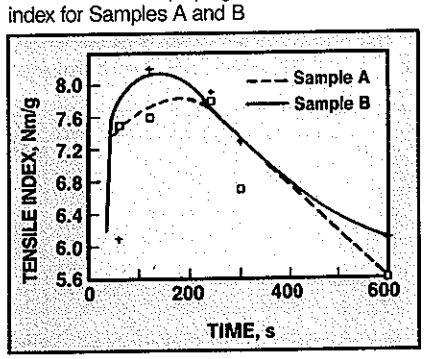
Surface properties

The pattern of fiber distribution in paper influences the appearance and physical properties (*4*). In papers manufactured from glass fibers, two considerations determine how well the fibers are distributed in the sheet. First, the fibers must be dispersed, as shown in Fig. 1.

Second, the flocculation of the dispersed, shorter fibers must be controlled. Fibers flocculate for two reasons. First, they can physically entangle with their neighbors to form a loose floc. Mason has described the tendency for flocs to continually form and disperse at rates that depend on the fluid dynamics of the piping and headbox system. The behavior of flocs also depends on fiber length, consistency, fiber flexibility, smoothness, and shape (*9*).

The second reason fibers flocculate is a chemical one. The distribution of electrical charges on fiber surfaces acquired when the fibers are submerged in water influences flocculation. Typically, glass fibers acquire a

10. The effect of pulping time on sheet tensile index for Samples A and B

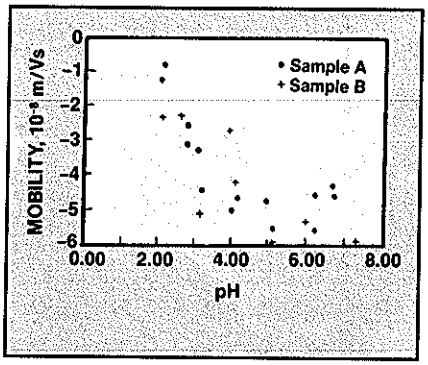


strong negative charge when submerged in water (*10*). In our study, we used electrophoretic mobility to characterize fiber charges and to relate those charges to paper properties.

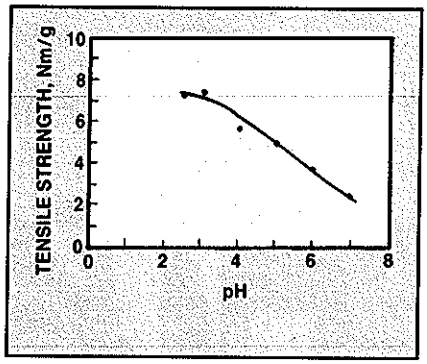
Generally, a population of highly charged particles or fibers will repel each other, while a reduction in the surface charge will permit attraction by van der Waals forces (*11*). Adjusting the pH is one way to influence surface charge and electrophoretic mobility. At low pH, the high proton concentration neutralizes the high electronegative charge at the surface of the fiber, and the electrophoretic mobility drops. For the glass fibers shown in **Fig. 11**, the isoelectric point (zero mobility) occurs between pH 1 and 2. The results in Fig. 11 show no obvious difference between the electrophoretic mobilities of Samples A and B, which indicates that the difference in sheet strength between the two is caused by differences in the physical properties of the fibers.

Handsheets were formed with Sample A fibers over the same pH range as the electrophoretic mobility measurements were made in Fig. 11. The tensile strength of these sheets is shown in **Fig. 12**. At low mobility (low pH), handsheets had greater strength

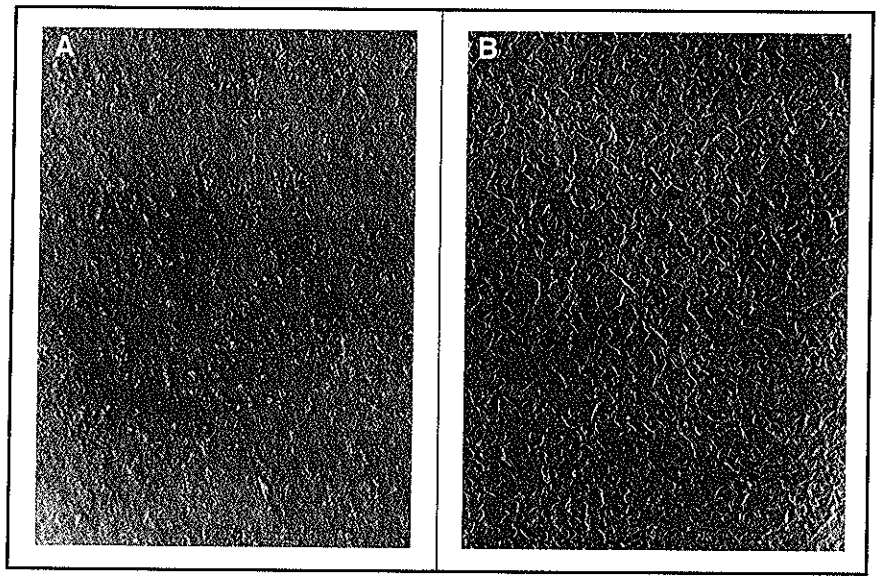
11. The effects of pH on the electrophoretic mobility of Samples A and B



12. The effect of pH on tensile index for Sample A



13. Photographs of the surface of sheets formed at pH 3 (A) and pH 5 (B)



and better formation than sheets formed at high pH, as shown in Fig. 12 and **Fig. 13.** At low mobility, fibers have a greater tendency to flocculate. However, the turbulence in sheet molds and commercial machinery is apparently sufficient to control the size and quantity of flocs and permit good formation and good strength.

The reasons for poor formation, shown in Fig. 13B, and low strength when formed at higher pH are not clear. It is commonly known that glass fibers leach in water. Scanning electron microscope photos taken of glass fibers that had been held at 65% relative humidity show obvious surface defects caused by moisture; these defects increase with time (*12*). It is possible that under some conditions, despite a high overall surface electronegativity, local positive and negative point charges may exist on the fiber surface. These may promote the flocculation that occurs at high pH (pH 4–7) shown in Fig. 13B.

A study of the surface of glass fibers after exposure to water between pH 3 and pH 7 might reveal differences in surface defects between fibers at these conditions. Further study of the composition of the pitted or leached areas described by Donnet (*12*) would indicate whether these areas could be the source of a electronegative charge different than the rest of the fiber surface. If so, this might explain the unexpected formation and tensile results observed at high pH values.

Summary

The physical dimensions and chemically induced properties of the surface of glass fibers influence final properties of a glass handsheet. Fiber length decreases with hydropulping time as fragile glass fibers break during dispersion. The sheet tensile strength increases with fiber hydropulping time because formation improves.

Extended pulping decreases mean fiber length and strength. The fiber diameter has an impact on fiber length since diameter influences the resistance of individual fibers to breakage during hydropulping. Direct measurements of length and diameter showed that both distributions were log normal, indicating that the numerous short fibers were the result of numerous fibers of small diameter.

Comparing direct diameter measurements with mean diameter determinations showed that some methods may not easily detect small differences in mean diameters or distributions. The examination of fiber interactions by electrophoretic mobility measurements indicated that changes in surface charges by pH adjustments have an impact on sheet formation and tensile strength.□

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