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# EVALUATION TECHNIQUES FOR FIBERS AND YARNS USED BY THE FIBROUS MATERIALS BRANCH, NONMETALLIC MATERIALS DIVISION, AIR FORCE MATERIALS LABORATORY

Compiled by the Fibrous Materials Branch

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AIR FORCE MATERIALS LABORATORY DIRECTORATE OF LABORATORIES AIR FORCE SYSTEMS COMMAND WRIGHT-PATTERSON AIR FORCE BASE, OHIO



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### FOREWORD

This report was prepared by the Fibrous Materials Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The work was initiated under Project No. 7320, "Fibrous Materials for Decelerators and Structures," Task 732001, "Organic and Inorganic Fibers."

This technical report has been reviewed and is approved.

in

H. ROSS, Acting Chief Fibrous Materials Branch Nonmetallic Materials Division Air Force Materials Laboratory

#### ABSTRACT

This report describes the techniques which are used by the Fibrous Materials Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, for evaluating some of the important mechanical and physical properties of fibers and yarns. These include density, strength, modulus of elasticity, and repeated flexing properties.

The test procedures described are those in use at the time this report was prepared. They have been in use for some time and a considerable amount of data have been generated which has demonstrated the reliability of the methods.

These techniques and procedures will be reviewed and updated as required. They are compiled and reported herein for the purpose of: (1) acquainting those concerned with fiber property measurement with those techniques which have been found to be useful for gathering data on a variety of types of fibers; and (2) to provide Air Force contractors and fiber users with the basic techniques used by the Air Force Materials Laboratory in order that valid comparisons of data can be made.

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# INTRODUCTION

This report describes the techniques which are used for evaluating certain of the mechanical properties of yarns and fibers in the Fibrous Materials Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio.

The fibrous materials, which are of interest to the Air Force, cover an extremely broad range of properties and dimensions. The individual fibers may have diameters ranging from 0.01 to 0.5 mils; lengths from a fraction of an inch to infinitely long; tensile strengths from  $10^3$  to  $10^6$  psi; rupture elongations from less than 1% to 50% or more; modulus values ranging from less than  $10^6$  to  $10^8$  psi; specific gravities from about 1 to almost 10; some may be very brittle, others very pliable.

It is apparent that the techniques used for evaluating the characteristics of such a broad range of materials must be carefully selected, and that any one technique may not be suitable for all fibrous materials requiring evaluation. Selection of the technique in any particular instance becomes an important consideration, therefore, particularly because the numerical value obtained for the property being measured may depend to a significant extent on the particular technique which is used.

The techniques described herein are those which have been in use for some time in the laboratory of the Fibrous Materials Branch of the Air Force Materials Laboratory. A considerable amount of data has been accumulated using these techniques. The use of techniques which deviate from those described herein can result in data which cannot easily be interpreted in relation to this accumulated information. It is important, therefore, that the characteristics of all fibrous materials intended for use by the Air Force be evaluated by the techniques outlined in this manual. No claim is made that these techniques are the only possible procedures which can provide meaningful data, but experience has proven them to be relatively straightforward, and sufficiently reliable for the purpose for which they were intended.

Procedures are described for evaluating such characteristics as tensile strength at temperatures up to 4000°F; elongation; tensile, torsional, bending and sonic modulus; flex resistance; specific gravity; cross-sectional area. The various instruments to be used are briefly described but specific instructions for instrument operation are not included except in those cases where the instrument operational manual does not provide instructions pertinent to the specialized technique being described. The proper method of recovering and recording the data is described in each case.

The test procedures described herein are those in use at the time of writing. This cannot be considered a static situation, for as new materials become available and the requirements for specialized testing change, the techniques will undoubtedly be modified, and new ones will be developed. In case of doubt, it would be wise to inquire of the Fibrous Materials Branch as to whether or not the present description of the test method is still applicable.

# METHOD FMT-1

# TENSILE TESTS OF FIBERS USING THE INSTRON TENSILE TESTER

#### 1. SCOPE

The techniques are suitable for the evaluation at room temperature of the tensile properties of all fibers which can be gripped by the methods described. Fiber lengths may be as short as 0.5 inch, though a 1-inch gage length is preferred. If an evaluation of initial modulus is needed, refer to Methods FMT-2, FMT-3, and FMT-8.

2. THE TENSILE TESTER

2.1 The techniques have been designed to be used with an Instron Tensile Tester. (See Figure 1.) However, other makes of tester may be used provided their characteristics conform to those of the Instron. The critical parameters are as follows:

- 2.1.1 Speed of the moving jaw
- 2.1.2 Speed of the recorder chart
- 2.1.3 Various full scale load ranges available
- 2.1.4 Maximum deflection of the "stationary" jaw

0.2 inch per minute (See Section 5)

20, 10 and 2 inches per minute

between 10 grams and 50 pounds

- 6 mils
- 2.1.5 The full scale response time of the recorder pen

not to exceed one second



Figure 1. Instron Tensile Tester

2.1.6 The recorder chart to be wide enough and the load measuring mechanism sensitive enough that load may be determined accurately to 1/200 of full scale or better.

2.1.7 The moving jaw to attain maximum speed from a stopped position of a time of not more than one second.

- Note 1: In the Instron tester three load cells may be used. The deflection of the loadmeasuring mechanisms in the A cell at maximum load of 50 grams is 5 mils; that of the B cell at maximum load of 2000 grams is 1.7 mils; that of the C cell at maximum load of 50 pounds is 5.2 mils.
- 3. PREPARATION AND CALIBRATION
- 3.1 Set the tensile tester to provide for the following operating conditions:

3.1.1	Crosshead speed	0.2 inch per minute (See Section 5)
3.1.2	Chart speed	20 inches per minute for fibers having a break- ing elongation of 10% or less
		10 inches per minute for fibers having a break-ing elongation of 10-30\%
		2 inches per minute for fibers having a break- ing elongation greater than 30%
3.1.3	Load cell	A for paper tab mounting
		B or C for other methods of mounting
3.1.4	Load range	Full scale load not more than twice the break- ing load of the specimen
3.1.5	Initial jaw separation	1 inch

3.2 Adjust and calibrate the tensile tester in accordance with the instructions supplied in the manufacturer's operating manual.

3.3 Adjust and calibrate the integrator, if energy is to be measured, in accordance with the instructions supplied in the manufacturer's operating manual.

#### 4. SPECIMEN MOUNTING

4.1 Direct Grip Jaws

4.1.1 Special rubber-lined jaws are available for all Instronload cells (see Figure 2). These may be used for any fiber which can be gripped adequately without damage. Evidence of unsatisfactory gripping may be (1) slippage in the jaws which is immediately visible on the chart trace (See Appendix III), (2) consistent breaking of the fiber adjacent to the edge of the jaw face which is an indication that the fiber has been damaged by the clamping action, (3) an excessive amount of extension of the fiber between the faces of the jaws known as jaw penetration (See Appendix V), (4) breakage of very brittle fibers due to crushing while the jaws are being tightened.

4.1.2 If any of these faults is detected, one of the other two methods of mounting should be used.





Figure 2. Direct Grip Rubber-Lined Jaws

4.2 Faper Tab

4.2.1 Any fiber whose diameter is not over one (1) mil and whose strength is not more than a few hundred grams can be tested by mounting on a specially-designed paper tab. The dimensions of the tab are illustrated in Figure 3. Mount the fiber along the axis of the slot using sealing wax as the adhesive.

4.2.2 Grip the fiber gently between thumb and finger and hold one of its ends against the paper in a position centrally located at one end of the slot and with the fiber axis parallel to the long edge of the paper tab. Dip the tip of a fine-tipped soldering iron into a dish of pulverized sealing wax and touch it quickly to the fiber so that a small drop of sealing wax falls on and surrounds the fiber and runs just up to the end of the slot. Allow a few seconds for the sealing wax to harden. Then grasp the other end of the fiber between thumb and finger and straighten the fiber so that it lies along the center of the slot. Apply another small drop of sealing wax to the other end of the fiber to the end of the slot.

4.2.3 Write any necessary fiber identification on the paper tab.

4.2.4 This method of mounting is particularly suitable for fine, delicate fibers, because it minimizes the likelihood of damage while the fibers are being handled, such as when measuring diameters, and mounted in the testing machine.

4.3 Epoxy Tab

4.3.1 This method of mounting is especially suitable for high modulus materials  $(>25 \times 10^{\circ})$  psi) or for fibers having a diameter of more than about 1 mil.



Figure 3. Paper Fab Fiber Mount

4.3.2 The epoxy which is used is a mixture of 40 parts by weight of General Mills Versamid 140 and 60 parts of Shell Chemical Company Epon 820.

4.3.3 Scribe two lines on a Teflon sheet exactly one inch apart. Using masking tape, attach pieces of wax paper to the Teflon sheet outside these lines so that the straight edges of the paper fall exactly along the lines. Hold the fiber with one end on each piece of wax paper so that it is straight and exactly perpendicular to the two parallel lines. Apply sufficient epoxy to each end of the fiber on top of the wax paper to form a tab having an area at least half an inch wide by one inch long (See Figure 4). Be sure that the epoxy runs right to the eage of the wax paper, but not beyond it, forming a well-defined straight edge. Allow the epoxy to cure overnight at room temperature.

Note 2: More rapid curing may be carried out at 175°F for two hours. However, this more rapid curing does not always permit air bubbles to rise to the surface of the epoxy so that inadequate adhesion sometimes results.

4.3.4 The epoxy does not adhere to the wax paper, so after hardening it may easily be lifted off. It is important to handle the fiber with extreme care, holding an epoxy tab in each hand, for the tabs are relatively heavy and indiscriminate handling could damage the fiber prior to testing. It is best to leave the fibers lying flat on the Teflon sheet until ready to mount them in the machine for testing.



Figure 4. Epoxy Tab Fiber Mount

### 5. RATE OF STRAIN

5.1 Most fibers can be tested at the rate of strain specified, i.e., 0.2 in/min. for a 1-inch specimen, or 20% of the specimen length per minute. The use of this rate, however, is based on the assumptions that:

5.1.1 The characteristics of the material being tested are not such that speed of response of the recorder is exceeded.

5.2.2 The characteristics of the material being tested are not speed-sensitive in this range of strain rates, that is, change of the order of a factor of 10 in the strain rate will not give significantly different results.

5.2 In any particular case one or both of these assumptions may be invalid. If the speed of response of the recorder is being exceeded, the strain rate must be reduced, or erroneous results will be obtained. If the material is known to be sensitive to changes in strain rate, consideration should be given to investigating the magnitude of the effect by testing at a range of speeds.

5.3 A method for determining whether the speed of response of the recorder is being exceeded is described in Appendix II. If the specimen cannot be tested at the strain rate specified, reduce the crosshead speed to 0.02 in/min or increase the specimen gage length, or both, to reduce the strain rate to a tolerable value.

# 6. TEST PROCEDURE

Note 3: It is extremely important, regardless of which method of fiber mounting is used, that when the fiber is mounted in the test machine the axis of the fiber be accurately parallel to the direction of application of load. If the fiber axis is not parallel to this direction, a shearing force and/or bending couple is applied during the test which, for some fibers, may significantly alter the apparent load-elongation behavior.

6.1 Direct Grip Jaws

6.1.1 Grasp the fiber gently between thumb and finger and, holding its axis parallel to the direction of application of load, insert one end of the fiber in the jaw attached to the load-measuring mechanism. Tighten the jaws onto the fiber sufficiently to prevent slippage during the test. (Note: In the A-cell jaws, type 2A, the clamping pressure is varied by changing the compression of the spring.)

6.1.2 Allow the fiber to hang freely through the opened pulling jaws. Grasp the fiber end below the pulling jaws and apply just enough tension to straighten the fiber. Tighten the pulling jaws sufficiently to prevent slippage during the course of the test.

- Note 4: It is sometimes helpful to activate the load-measuring mechanism while the fiber is being gripped in the lower jaws. Apply sufficient tension to cause the recorder pen to move slightly away from the zero load axis.
- 6.2 Paper Tab

6.2.1 For fibers which have been cemented to paper tabs, use Instron A cell rubber-lined plastic grips. Clamp one end of the paper tab in the jaws attached to the load-measuring mechanism, being careful to hold the tab so that the axis of the fiber is parallel to the direction of application of load. Allow the other end of the paper tab to fall between the open pulling jaws and tighten these jaws on the paper.

Note 5: In this case the distance between the jaws should be raised to 1-1/2 inches. The test gage length is defined by the free length of fiber between opposite ends of the slot. It is not necessary to grasp the sealing wax in either jaw.

6.2.2 With a sharp pair of scissors, carefully cut a 1/8-inch slot out of one side of the paper tab. Grasp the two cut ends of paper between the thumb and finger of one hand and carefully cut a 1/8-inch slot out of the other side of the tab (See Figure 5). The fiber will now be the sole remaining connection between the pulling jaw and the load-measuring jaw.

6.3 Epoxy Tab

6.3.1 Grip the epoxy tab between the rubber-faced jaws set to an initial spacing of 1-1/2 inches. In general, it will only be possible to use the B or C cell jaws for this purpose, eliminating the possibility of using the A cell.

6.3.2 Handle the fiber with extreme care because of the mass of the epoxy tabs.

6.3.3 Grip one epoxy tab in the jaws attached to the load-measuring mechanism, being careful that the fiber is not bent at the point where it emerges from the epoxy when the free fiber axis is parallel to the direction of application of load. Allow about 1/4 inch of the tab to protrude below the edge of the jaw faces (See Figure 6).



Figure 5. Cutting Paper Tab for Testing

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Figure 6. Epoxy Tab Mounted in Direct Grip Jaws

6.3.4 Allow the other epoxy tab to hang between the open pulling jaws, again being careful that the fiber is not bent at its point of emergence from the epoxy, and tighten the pulling jaws on the epoxy tab.

- Note 6: It is extremely important that the fiber be absolutely straight from its point of embedment in one epoxy tab through the unsupported length of the fiber to the embedment in the other epoxy tab. Any deviation from a straight line may significantly affect the load-elongation behavior.
- 7. RESULTS
- 7.1 Tensile Strength

7.1.1 Determine the breaking load of each fiber as the maximum load which has been encountered between the start of the test and the final rupture. Read this load with an accuracy corresponding to 1/500 of the full scale load of the chart.

7.1.2 If the fiber extends a significant amount after the maximum load has been reached, quote also the load at rupture, which is the load at the point where the fiber finally parts and the load falls quickly to zero. In any case of unusual stress-strain behavior, that is, when the rupture load is significantly different from the breaking load or when no clearcut rupture occurs, include a typical stress-strain curve in the report.

7.1.3 Report:

7.1.3.1 The type of fiber tested

7.1.3.2 The average diameter of the fibers tested, in mils.

7.1.3.3 The type of mount used

7.1.3.4 The number of fibers tested

7.1.3.5 The average breaking load in grams to three significant figures, and the range covered by the individual results.

7.1.3.6 The average load to rupture, if this was different from the breaking load, and the range covered by the individual results.

7.1.3.7 The standard deviation of individual values of fiber breaking load and rupture load, if desired.

Note 7: This may be obtained from the range of the measurements in each case.

7.1.3.8 The tensile strength in  $lb/inch^2$  quoted to three significant figures only (See Appendix VII).

Note 8: This is calculated from measurements of breaking load and average diameter.

7.2 Breaking Elongation

7.2.1 For each fiber, read the breaking elongation (i.e., the elongation corresponding to the breaking load) to the nearest 0.01 inch of extension. Express this as a percentage of the initial gage length of the specimen.

7.2.2 Report:

7.2.2.1 The type of fiber tested

7.2.2.2 The number of fibers tested

7.2.2.3 The average breaking elongation and range of individual results to the nearest 0.1%.

7.2.2.4 The standard deviation of breaking elongations of individual fibers, if desired.

Note 9: This may be obtained from a range of the individual measurements.

7.3 Energy to Rupture

7.3.1 For most fibers, the energy to rupture may be measured directly during the course of the test by using the Instron integrator. Complete instructions for the use of this device are given in the manufacturer's operating manual.

7.3.2 When the integrator has been calibrated according to the instructions in the manual, calculate the energy from the integrator reading as follows:

$$W = \frac{X}{5000}$$
. L . S

where

- W = energy in inch-pounds
- X = integrator reading L = full scale load in pounds
- S = crosshead speed in inches/min

7.3.3 When the point of rupture is not the same as the point at which maximum load occurs, or when a clearcut rupture does not take place, the integrator can only be used when it is stopped manually by turning off the switch as soon as the load begins to drop. Often in such a case, or in any other case where the integrator is not available, it is best to obtain the energy to break the specimen directly from the chart by using a planimeter, or other means of determining the area under the curve. Any planimeter capable of measuring this area accurately to the nearest 0.01 square inch may be used. Measure the area under the curve as follows:

7.3.3.1 Draw a line perpendicular to the zero load axis through the point at which maximum load occurs. The area to be measured lies between this line, the zero load axis, and the load-elongation curve down to the point at which the test commenced. Traverse this area with the planimeter and calculate the average area measurements obtained from each curve in square inches.

Note 10: If a planimeter is not available, a sufficiently accurate measure of this area may be obtained by carefully cutting out the chart delineated by the zero load axis, the vertical line at the point of maximum load, and the load-elongation curve from the start of the test. Weigh this piece of paper and work out its area in square inches from the weight per unit area of the chart paper.

7.3.4 Report:

7.3.4.1 The type of fiber tested

7.3.4.2 The number of fibers tested

7.3.4.3 The average energy to rupture, and the range of individual values expressed in inch-lb per inch of gage length.

7.3.4.4 The standard deviation of individual values, if desired.

#### 8. CALCULATIONS

Symbol	Quantity	Units	How Measured
s <sub>T</sub>	tensile s <b>trength</b>	pounds per square inch (psi) grams per denier (gpd) grams per tex (gpt)	FMT-1, 3, 4, 14
$^{L}B$	breaking load	pounds, grams	FMT-1, 3, 4, 14
е <sub>В</sub>	breaking elonga- tion	%	FMT-1, 3, 14
w <sub>B</sub>	work or energy to break	inch pounds/inch	FMT-1
d	fiber diameter	mils	FMT-6

A	cross-sectional area	square mils	FMT-6
D	linear density	denier	FMT-5
0	density	grams per cc:	FMT-7
М	magnification factor	chart speed (inches/min) speed of pulling jaw (inches/min)	
G	initial gage length	inches	
Calculate	From	Formula	
s <sub>T</sub>	L <sub>B</sub> , A	$S_T = \frac{L_B}{A} \times k$ where $k = 10^6$ for $Z$	$L_{\rm B}$ in pounds
	L <sub>B</sub> , d	$S_{T} = \frac{L_{B}}{d^{2}} \times k$ where k = 1.27 x 1 = 2.81 x 1	$0^6$ for L <sub>B</sub> in grams $0^3$ for L, in grams
	gpd, $\rho$	$S_{T} = gpd \times \rho \times 1.28 \times 10^{4}$	ВС
S <sub>T</sub> gpd	L <sub>B</sub> , D	$S_{T} = \frac{L_{B}}{D} x k where k = 1 \text{ for } L_{B}$ = 454 for 1	in grams
	psi,ρ	$S_{T} = psi x \frac{1}{\rho} x 7.81 x 10^{-5}$	в
S <sub>T</sub> gpt	gpd	$S_{T} = 9 x gpd$	
	psi, p	$S_{T} = psi x \frac{1}{\rho} x 7.03 x 10^{-4}$	
<sup>е</sup> в%	chart inches, [M, G]	$e_{B} = \frac{\text{inches of chart}}{G} \times \frac{1}{M} \times 100\%$	
W <sub>B</sub> in.lb/in.	Instron inte- grator read- ing, G	$W_{B} = \frac{\text{integrator reading}}{5000} \text{ x full sca}$ $x \frac{\text{speed of pulling jaw in./mi}}{1000}$	nle load (lb) n
	planimeter, C,M (Instron Chart)	$W_{\rm B} = \frac{\text{area (in.}^2)}{G} \frac{\text{full scale load}}{9.53}$	$\frac{\text{(b)}}{M} \times \frac{1}{M}$

9. EXAMPLE		
If		$L_B = 15.5$ g on a full scale of 50 g = 0.034 lb on a full scale of 0.1 lb
		d = 0.55 mil
		A = 0.238 square mils (see Appendix VII)
		$\rho = 2.49 \text{ g/cm}^3$
		D = 3.5 denier
		G = 1.0 inch
	j	aw speed = 0.2 inches/min
	cha	art speed = $20$ inches/min
		$M = \frac{20}{0.2} = 100$
	chart inches	to break = 1.34 inch
	integrator	reading = 59
	planimeter	reading = $2.17$ inches <sup>2</sup>
Calculate	From	Results
S <sub>T</sub> psi	L <sub>B</sub> , A	$S_{T} = \frac{0.034}{0.238} \times 10^{6} = 1.4 \times 10^{5} \text{ psi}$
		or S <sub>T</sub> = $\frac{15.5}{0.238}$ x 2.205 x 10 <sup>3</sup> = 1.4 x 10 <sup>5</sup> psi
	L <sub>B</sub> , d	$S_{T} = \frac{0.034}{(0.55)^2} \times 1.27 \times 10^{6} = 1.4 \times 10^{5} \text{ psi}$
		${}^{\text{or}}{}_{\text{T}} = \frac{0.034}{(0.55)^2} \times 2.81 \times 10^3 = 1.4 \times 10^5 \text{ psi}$
S <sub>T</sub> gpd	L <sub>B</sub> , D	$S_{T} = \frac{15.5}{3.5} = 4.4 \text{ gpd}$
		or $S_{T} = \frac{0.034}{3.5} \times 454 = 4.4 \text{ gpd}$
	psi <b>, p</b>	$S_{T} = \frac{1.4 \text{ x}}{2.49} \frac{10^{5}}{2} \text{ x} \ 7.81 \text{ x} \ 10^{-5} = 4.4 \text{ gpd}$

Calculate	From	Results (Cont.)
<sup>S</sup> T bai	gpc, p	$S_{T} = 4.4 \times 2.49 \times 1.28 \times 10^{4} = 1.4 \times 10^{5} \text{ psi}$
S <sub>T</sub> gpt	gpd	$S_{T} = 9 x 4.4 = 40 gpt$
	psi, p	$S_{T} = \frac{1.4 \times 10^{5}}{2.49} \times 7.03 \times 10^{-4} = 40 \text{ gpt}$
e <sub>B</sub> %	chart inches, M, G	$e_B = \frac{1.34}{1.0} \times \frac{1}{100} \times 100\% = 1.3\%$
W <sub>B</sub> in.lb./in.	integrator reading, G	$W_{B} = \frac{59}{5000} \times 0.1 \times \frac{0.2}{1.0} = 2.4 \times 10^{-4}$ in lb/in.
	planimeter, G, M (Instron chart)	$W_{B} = \frac{2.17}{1.0} \times \frac{0.1}{9.53} \times \frac{1}{100} = 2.3 \times 10^{-4}$ in lb./in.

### METHOD FMT-2

# TENSILE RECOVERY, CYCLING AND HYSTERESIS MEASUREMENTS USING THE INSTRON TESTER

### 1. INTRODUCTION

The performance in service of structures like fabrics, webbing, tapes, etc., which are made of fibrous materials, is often influenced by the ability of the structure to recover its original dimensions after the application of stress. This ability to recover is dependent in part upon the geometry of the structure itself, and upon the degree and kinds of interaction which exists between the fibers within that structure, but it is also strongly dependent upon the elastic properties of these component fibers, and particularly upon their ability to recover from imposed strain. Mecsurements of tensile recovery, therefore, both in terms of dimensional recovery and energy loss, can be an important characteristic in evaluating the potential of any particular fiber for a variety of end uses. Several measurements which can be made on fibers or on yarns are described below.

# 2. PREPARATION

2.1 These tests can be carried out under the same conditions as the tensile tests described in Methods FMT-2 and FMT-14. The same methods of specimen mounting are suitable. A gage length of either one inch or ten inches can be used. The speeds of operation can be the same as those used in a normal tensile test.

2.2 Recovery measurements will usually be made after the specimen has been extended to a specified load or a specified strain. The Instron tester can be set up so that the crosshead reverses its direction of travel when the load on the specimen has built up to a preset amount, or when the extension has reached a preset value. Instructions for operating the load or extension cycling controls are given in the Instron instruction manual.

# 3. CHOICE OF LIMITS

3.1 The limits of stress or strain from which recovery is to be measured cannot be assigned arbitrarily for any given specimen, but must be selected in consideration of the intended end use. If the end use is to be space suits, for example, recovery from relatively low strain levels becomes important. Fiber strains which will be encountered, for example, in a fold, will in general be less than about 5%. Strain levels as high as 10 to 20% may be encountered in a very sharp crease or in the stretching which may occur at a bent elbow or knee. Fibers which are intended for use in applications such as parachute canopies or risers, on the other hand, will encounter levels of stress application which depend upon the safety factor which has been designed into the structure. For such applications, it is best to select a level from which recovery is to be measured in terms of the design safety factor and the breaking load of the fiber.

3.2 Some typical choices of end points from which recovery should be measured are listed in Table I.

#### 4. PROCEDURE

4.1 Adjust the load or extension cycling controls on the Instron to reverse the direction of motion of the crosshead at the selected value of load or extension.

# TABLE I

### END POINTS FOR RECOVERY TESTS

End Use

Cycled to

space suits, etc.

2%, 5%, 10%, 20% strain

other -

design safety factor 5 or more

design safety factor 2 to 4.9

design safety factor less than 2

10%, 20%, 50% of breaking load

20%, 50%, 75% of breaking load

50%, 75%, 90% of breaking load

4.2 Set the Instron integrator on AUTOMATIC OPERATION so that the areas under both extension and recovery curves will be recorded.

Note 1. When the switch on the integrator is in the AUTOMATIC position, one counter will stop operating and the other start at the instant when the crosshead reverses its direction of travel.

4.3 Mount the specimen in the jaws of the Instron as outlined in Method FMT-1, and operate the Instron so that a complete extension-recovery cycle is recorded. Stop the crosshead when it has returned to the position corresponding to the jaw separation at the start of the test.

4.4 Start a stop-watch at the instant the crosshead stops moving. Allow the specimen to recover for five minutes.

4.5 During this five-minute period, read the two counters on the Instron integrator and turn the integrator off.

4.6 After five minutes of recovery, start the crosshead again in a downwards direction. Stop it when the load-elongation curve has departed appreciably from the zero-load axis.

4.7 Under some circumstances, it may be desirable to carry the specimen through several cycles of elongation and recovery. This should be done by reversing the direction of motion of the crosshead as soon as it returns to the position corresponding to the initial gauge length separation of the jaws, without permitting five minutes recovery between cycles. Usually it will be found that the unrecovered extension will gradually increase through a few cycles of extension and recovery, but that these cycles will become reproducible after about four or five have been carried out. This is a reasonably rapid way of determining the maximum amount of unrecovered extension which may be expected. It is still desirable at the end of the final cycle to permit five minutes of recovery in order to obtain an estimate of the magnitude of delayed recovery which may be expected.

5. RESULTS

5.1 Several parameters which are helpful in predicting how any fiber will perform in service may be obtained from this type of elongation-recovery test. Of particular interest are the

energy recovery and the percent immediate and delayed strain recovery. The method by which these quantities are calculated is outlined in Appendix II and a numerical example is given in Section 7 for illustration.

5.2 Report the following:

5.2.1 Type of fiber or yarn tested

5.2.2 Number of specimens

5.2.3 The quantities which have been calculated, giving both the average and the range of values obtained.

5.2.4 The standard deviation of the individual values for each quantity measured, if desired.

6. CALCULATIONS

See Figure 7.

Any of the following quantities may be of interest:

Imposed strain = 
$$\frac{OP/M}{\ell g} \times 100\%$$
  
Recovered strain (immediate) =  $\frac{BP/M}{\ell g} \times 100\%$   
Unrecovered strain (immediate) =  $\frac{OB/M}{\ell g} \times 100\%$   
Recovered strain (delayed) =  $\frac{BC/M}{\ell g} \times 100\%$   
Unrecovered strain (permanent) =  $\frac{OC/M}{\ell g} \times 100\%$   
% Surain recovery (immediate) =  $\frac{BP}{OP} \times 100\%$   
% Strain recovery (delayed) =  $\frac{BP}{OP} \times 100\%$   
% Energy recovery\* =  $\frac{Area BRAP}{Area OEAP} \times 100\%$ 

# 7. EXAMPLE

If

$$OP = 5.00$$
 inches  
 $BP = 3.16$  inches

\* Often called resilience, and expressed as a decimal fraction.



Figure 7. Parameters Derived from Elongation- Recovery Curves

- OEA = First Extension Curve
- ARB = Recovery Curve
  - CD = Second Extension Curve
- Area OEAP = Energy to Extend (in-lb)
- Area BRAP = Energy recovered (in-lb)
  - OB = "Immediate" unrecovered length (in.)
  - BC = Recovery in time t under zero load = "delayed" recovery (in)
  - OC = "Permanent" unrecovered length (in)
  - lg = Initial gage length (in)

OB = 1.84 inches BC = 0.93 inches OC = 0.91 inches  $\ell g = 1.0$  inches jaw speed = 0.2 inches/min

chart speed = 10 inches/min

$$M = \frac{10}{0.2} = 50$$

Area OEAP = 14.22 square inches Area BRAP = 5.27 square inches

Then

Imposed strain = 
$$\frac{5.0}{50 \text{ x } 1.0} \text{ x } 100\% = 10.0\%$$
  
Recovered strain (immediate) =  $\frac{3.16}{50 \text{ x } 1.0} \text{ x } 100\% = 6.3\%$   
Unrecovered strain (immediate) =  $\frac{1.84}{50 \text{ x } 1.0} \text{ x } 100\% = 3.7\%$   
Recovered strain (delayed) =  $\frac{0.93}{50 \text{ x } 1.0} \text{ x } 100\% = 1.9\%$   
Unrecovered strain (permanent) =  $\frac{0.91}{50 \text{ x } 1.0} \text{ x } 100\% = 1.8\%$   
% strain recovery (immediate) =  $\frac{3.16}{5.00} \text{ x } 100\% = 63\%$   
% energy recovery =  $\frac{5.27}{14.22} \text{ x } 100\% = 37\%$ 

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# METHOD FMT-3

# TENSILE TESTS OF FIBERS USING THE OPTICAL STRAIN ANALYZER

# 1. DESCRIPTION OF APPARATUS

1.1 The optical strain analyzer and its use are described in detail in Technical Reports AFML-TR-65-47 dated February 1965, and AFML-TR-65-366 dated December 1965. The unique characteristics of this instrument are: (a) grips capable of holding even extremely brittle fibers without damage; (b) an optical tracking system which follows two flags hung on the specimen at a known initial separation, permitting extremely accurate measurement of elongation up to a limit of 100 mils; (c) measurement at temperatures up to 3500°F in an inert gas atmosphere.

1.2 The complete apparatus is shown in Figure 8. The specimen is held horizontally in jaws mounted on a screw-driven strain mechanism. Load is measured by strain gauges on parallel flat spring load cells that support the stationary grip. The grips are lined with two flat pieces of aluminum (for room temperature testing) or tantalum (for elevated temperatures) arranged to meet at a slight angle (See Figure 9). The liners are softer than the fiber, and are deformed when the fiber is clamped, the area of contact being greatest toward the outer end of the fiber and gradually decreasing to zero about half way across the liner. The clamping pressure is thus free of sharp gradients that might cause premature failure of brittle fibers. A small clamping force is applied initially by springs, and the linkage on the grip arms is arranged to increase this initial force in proportion to the pulling load. Each fiber specimen is clamped between fresh liner surfaces; the liners are changed after three uses.



Figure 8. Optical Strain Analyzer





Figure 9. Gripping Action in Optical Strain Analyzer

1.3 Strain is measured by an optical extensioneter which tracks the silhouette of two gage markers hung on the specimen. The gage markers are bent in the form of a  $\mu$  and are hung on the specimen at an initial separation of between 1/4 and 1 inch. Back lighting provides a silhouette onto which the photodetectors are locked. Any movement of either marker is followed by a similar movement of the corresponding photodetector so that the distance between the photodetectors is an accurate measure of the elongation of the specimen. This elongation can be recorded with an uncertainty due to machine noise of less than 20 microinches.

1.4 Elevated temperatures are provided by a split graphite tube furnace capable of operating at temperatures up to 3500°F. Above 2500°F the markers which are hung on the specimen become sufficiently self-illuminating to be detected directly by the photocells without any background illumination. Grips for elevated temperature operation are made of graphite and use tantalum liners. The entire gripping system is enclosed by the furnace and a large disc placed at the base of each grip pull rod dissipates the heat providing protection for the load cell and crosshead drive system.

1.5 The crosshead speed can be varied between 4 mils and 2 inches per minute. Five interchangeable strain gage load cells provide a load measuring range of 25 grams to 50 pounds.

2. USE OF THE OPTICAL STRAIN ANALYZER

2.1 This instrument should be used whenever very accurate measures of elongation or modulus are desired, and with fibers which are too brittle to be tested by the techniques described for use with the Instron tester.

2.2 Detailed operating instructions are available in the manual supplied with the instrument. A sample load-elongation curve derived from the instrument is shown in Figure 10.





3. REPORTS

Report the following information:

- 3.1 Type of fiber tested.
- 3.2 Average diameter of fiber in mils.
- 3.3 Number of tests.
- 3.4 Operating temperature
- 3.5 Average breaking load and range of individual values.
- 3.6 Standard deviation of the breaking load for individual fibers, if desired.
- 3.7 Tensile strength in psi given to three significant figures.
- 3.8 Average elongation to break in percent and range of individual values.
- 3.9 Standard deviation of elongation to break for individual fibers, if desired.
- 3.10 Average initial modulus in psi and range of individual values.
- 3.11 Standard deviation of initial modulus for individual fibers, if desired.
- 3.12 A sample load-elongation curve.

### METHOD FMT-4

# MEASUREMENT OF TENSILE STRENGTH OF FIBERS AT ELEVATED TEMPERATURES USING THE DUAL HEAD TENSILE TESTER

# 1. DESCRIPTION OF TI VISTRUMENT

This instrument (See Figure 11) is designed to test any fiber having a length of at least four inches and a diameter of not more than about two mils. The fiber specimen is mounted on two flat half-jaws coated with sealing wax which can be melted by an integral electrical resistance coil. The room temperature gage length is about 2-3/4 inches, of which the central one-inch portion can be heated in a split induction furnace capable of temperatures up to 2000°F. The speed of motion of the moving jaw can be varied continuously from zero to a maximum of 2.07 inches per minute. Two load ranges are available, 0 to 250 grams and 0 to 10 pounds. The output of the dynamometers is plotted on a Model 906C Honeywell Visicorder.

# 2. OPERATION OF THE INSTRUMENT

General operating instructions are given in the manufacturer's operating manual. Specific instructions are given below.

# 2.1 Speed of Motion of the Moving Jaw

Set the moving jaw to move at a speed of 0.44 inch per minute. Because the crosshead speed control dial is not calibrated directly in speed of motion of the jaw, the following calibration procedure must be used.

2.1.1 Mount the dial indicator to the frame of the instrument so that its foot bears on the end of the crosshead opposite to that carrying the jaw (See Figure 12).



Figure 11. Dual Head Tensile Tester



Figure 12. Operating Speed Calibration -- Dual Head Tensile Tester

2.1.2 Adjust the recorder pen to read 0 for the at-rest position of the crosshead.

2.1.3 Set the crosshead speed control dial for a relatively slow speed of motion of the crosshead.

2.1.4 Move the crosshead through a distance of exactly one inch as indicated by the dial gage.

2.1.5 Adjust the amplifier gain to give full scale deflection of the pen on the chart corresponding to one inch of crosshead motion.

2.1.6 Remove the dial gage and move the crosshead back to its zero position.

2.1.7 For a series of crosshead speed selector dial settings, move the crosshead through a distance of approximately one inch and obtain a series of lines plotted on the chart.

2.1.8 The actual speed of the crosshead corresponding to each of the crosshead speed control dial settings is obtained by measuring the slope of the lines on the chart corresponding to each of these settings.

2.1.9 Plot a calibration curve of crosshead speed against calibration dial control setting.

2.1.10 Use this calibration chart to set the crosshead speed control dial to give a crosshead speed of exactly 0.44 inch per minute.

2.1.11 Re-check a few points on the crosshead speed calibration curve periodically to make sure the speed settings remain accurate.

2.2 Fiber Mounting

2.2.1 Heat the right-hand mounting plate.

2.2.2 Form a loop one one end of the fiber, and hold the fiber in the left hand so that about 1/2 inch of the loop projects beyond the fingers. Lay the loop into the melted sealing wax on the right-hand mounting plate so that the long end of the loop is located exactly collinear with the axis of the susceptor.

2.2.3 Allow the sealing wax to cool and harden.

2.2.4 Hold the opposite end of the fiber in the left hand so that the fiber is carefully aligned on the susceptor axis and its end lies across the left-hand mounting plate.

2.2.5 Heat the left-hand mounting plate.

2.2.6 Allow the sealing wax to harden, embedding the fiber (See Figure 13).



Figure 13. Fiber Mounted in Dual Head Tensile Tester, Showing Susceptor

2.3 Operating Instructions

2.3.1 If the test is to be done at room temperature, follow the instructions given in the operating manual.

2.3.2 If the test is to be done at an elevated temperature, heat the susceptor to the desired temperature according to the instructions in the operating manual. Allow the fiber to warm up for the desired length of time, not less than one minute, after the susceptor reaches operating temperature before carrying out the test.

#### 3. REPORTS

Report the following information:

3.1 Type of fiber used.

- 3.2 Average fiber diameter, in mils.
- 3.3 Number of fibers tested.

3.4 Operating temperature.

3.5 Average breaking load to the nearest 0.1 gram if 250-gram dynamometer was used; or the nearest 0.01 pound if the 10-pound dynamometer was used, and the range of the individual values obtained.

3.6 Standard deviation of the individual test results, if desired.

#### METHOD F'MT-5

# MEASUREMENT OF LINEAR DENSITY

### 1. INTRODUCTION

1.1 The linear density of single fibers is commonly expressed in denier units. Denier is the weight in grams of 9,000 meters of material.

1.2 Three techniques are available for measuring linear density. They are described separately herein.

#### A MICROBALANCE

2. DESCRIPTION OF APPARATUS

2.1 A Mettler microbalance is used, having a sensitivity of about two micrograms.

#### 3. APPLICABILITY

3.1 This may be used whenever the total weight of the specimen is not less than 100 micrograms. This corresponds to the weight of 100 centimeters (about 40 inches) of fiber of diameter 10 microns (about 0.5 mil), and having a specific gravity of 1.

### 4. PROCEDURE

4.1 Weigh a measured length, which for ease in calculation should be 9 or 90 centimeters, for the determination of denier.

4.1.1 Calculate the linear density as follows:

Denier = 
$$9 \times 10^5 \frac{\text{w}}{\ell}$$

where w = weight in grams of  $\ell_{\rm cm}$  of fiber.

#### B. DENIER BALANCE

### 5. DESCRIPTION OF APPARATUS

5.1 The denier balance consists of the torsion balance reading directly in denier when a 90 cm length of fiber is weighed. Cross-sectional area can be calculated from the denier so obtained as outlined above.
## C. VIBROSCOPE

#### 6. DESCRIPTION OF APPARATUS

6.1 in the vibroscope a measured length of fiber is suspended under a known tension and caused to vibrate so that its resonant frequency can be determined. The relationship between linear density, specimen length  $\boldsymbol{l}$  in cm, tension T in g force, and resonant frequency f in cps, is:

Linear density = 2.21 x 
$$10^8$$
 x  $\frac{T}{\ell^2 f^2}$  denier

6.2 The vibroscope can be used with fibers having a linear density of not more than about 40-50 denier. It will be less effective for stiff materials than for flexible materials. It is inadvisable to use the vibroscope for highly conducting materials because of the possibility of short-circuiting the high-voltage exciting coils.

#### 7. OPERATING PROCEDURE

7.1 The method of using the vibroscope is described very completely in ASTM Method D1577-64T.

#### 8. CALCULATIONS

Symbol	Quantity	Units
D L W T f	linear density specimen length specimen weight tensioning weight resonant frequency	denier cm grams grams cycles/sec
Calculate	From	Formula
D	w, e	$D = 9 \times 10^5 \frac{w}{\ell}$
D	<i>l</i> , T, f	$D = 2.21 \times 10^8 \frac{T}{\ell^2 f^2}$
9. EXAMP	LE	$= \frac{3.31 \times 10^7}{\ell^2 f^2} \text{ when } T = 0.150 \text{ g}$
9.1 H, UBIN	g a Mettler balance,	w - 5.54 x 10 g
		$\mathcal{L}$ 90 cm $-4$
then		D = 9 x 10 <sup>5</sup> x $\frac{3.54 \times 10^{-1}}{90}$ = 3.5 denier
9.2 If, usin	g a vibroscope,	$\ell$ = 5.06 cm
		$\mathbf{T} = 0.150 \mathbf{g}$
		f = 600 cps
then		$D = \frac{3.31 \times 10^7}{5.06^2 \times 600^2} = 3.59 \text{ denier}$

## METHOD FMT-6

## MEASUREMENT OF FIBER DIAMETER AND CROSS-SECTIONAL AREA

#### 1. INTRODUCTION

The diameter of fibers having circular cross-section may be measured directly with a microscope using a calibrated eyepiece. Fibers which have elliptical cross-sections or irregular cross-sections such as graphitized viscose must be measured by cutting a cross-section and measuring the area of the projected image of the cross-section by a means of a planimeter. Both methods are described.

#### A. MEASUREMENT OF DIAMETER

#### 2. SCOPE

This method of measurement can be used for loose individual fibers or for fibers already attached to tabs for tensile testing, provided they have a circular cross-section. In the latter case, when each fiber is individually identified and measured, a more accurate value of tensile strength for each fiber can be calculated. This reduces considerably the variability between specimens and permits a more reliable estimate of tensile strength to be obtained.

#### 3. MEASURING EYEPIECES

3.1 Two types of measuring eyepieces are available for use with the microscope.

#### 3.1.1 Filar Micrometer Eyepiece

This eyepiece contains a scale which runs from 0 to 1000. The hairline can be moved across the scale by rotating a knob which carries a drum subdivided into 100 divisions around the circumference. One full rotation of the drum corresponds to one division on the eyepiece scale, which is 10 divisions long. The measurement is made by rotating the drum until the hairline just touches one side of the fiber image, reading the position of the hairline on the eyepiece scale and micrometer drum, and then moving the hairline by rotating the drum until it just touches the other side of the fiber (See Figure 14). A second reading of the position of the hairline on the eyepiece scale and micrometer drum setting is made and the difference between the two readings is a measure of the diameter of the fiber. This can be converted to microns or mils by proper calibration of the eyepiece scale, as described in Section 5.

#### 3.1.2 Image-Splitting Eyepiece (Cook - A.E.I)

This eyepiece provides greater sensitivity than the Filar, can be used with moving images, and does not require the subtraction of two readings. It produces two images of the fiber, one of which is red and the other, green. The position of these images relative to one another can be changed by rotation of the micrometer drum. When the images fall on top of one another so that the fiber looks black, the micrometer drum reads 0. The reading of the micrometer drum, when the images have been displaced until they just touch one another, is a measure of the diameter of the fiber (See Figure 15). Displace one image relative to the other in both directions, by rotating the drum both positively and negatively to obtain two measures of fiber diameter which can be averaged. The micrometer drum reading is converted to microns by a calibration procedure described in Section 5.



Figure 14. Filar Micrometer Eyepiece



#### 4. PROCEDURE

Note: Significant errors can be made in the measurement of fiber diameter by these techniques unless the microscope is properly adjusted. Because this is such a critical part of the procedure, adjustment of the microscope is outlined in detail. Any departure from the procedure outlined can result in a significant error in the calculation of cross-sectional area.

4.1 Insert the measuring eyepiece into the tube of the microscope, making sure that it is properly seated.

4.2 In the case of the Filar micrometer eyepiece, bring the hairline and eyepiece scale into sharp locus by rotating the knurled ring on the eyepiece itself. Turn the measuring drum until it reads 0. Look into the eyepiece to see whether the hairline falls directly on one of the major divisions of the eyepiece scale (0, 1, 2, 3, etc.). If this is not the case, hold the ring of the measuring drum which carries the scale with one hand, and rotate the knurled knob with the other until the hairline falls exactly on the top of one of the major eyepiece scale divisions. The hairline will now move from one major division to the next division on the eyepiece scale when the drum is rotated through one complete revolution, corresponding to 100 divisions on its circumferential scale.

4.3 Place the specimen on a microscope slide on the stage of the microscope. Select an objective lens to provide a magnification for which the fiber fills as much of the field as possible, but not so large that both edges of the fiber cannot be brought into relatively sharp focus at the same time. At no time should an objective lens having a magnification of more than about 40X be used, unless a mounting medium is also used.

4.4 Remove the eyepiece and look down the tube of the microscope. Center the light in the tube by adjustment of the light and the reflecting mirror at the base of the microscope. (Make sure the light is reflected off the plane surface of the mirror.) Replace the eyepiece, being careful that it is properly seated.

4.5 Carefully adjust the microscope for critical illumination as follows:

4.5.1 Half close the iris diaphragm in front of the light source. (Known as the field diaphragm.)

4.5.2 Rack the substage condenser up until it nearly touches the bottom of the microscope slide.

4.5.3 Open the substage diaphragm to its full extent.

- 4.5.4 Bring the fiber into sharp focus.
- 4.5.5 Close the substage diaphragm.

4.5.6 Move the lamp condenser back and forth until a sharp image of the light source is obtained on the substage diaphragm. (This may be made easier by inserting a white card in the plane of the diaphragm.)

Note 2: The above procedure is suitable for a light source having a ribbon filament. If the lamp has a straight or coiled wire filament, place a diffuser in front of it so that the illumination of the substage diaphragm is completely uniform. A piece of finely ground glass or very uniform paper is suitable for such a purpose.

4.5.7 Open the substage diaphragm to its full extent.

4.5.8 Check that the fiber is still in sharp focus.

4.5.9 Move the substage condenser until the circle of light limited by the field diaphragm is in focus.

Note 3: If the microscope light is not provided with a field diaphragm, hold a needle or pencil point immediately in front of the lamp condenser. Move the point back and forth and rack the substage condenser until the shadow of the moving point is in sharp focus.

4.5.10 Adjust the field diaphragm so that the circle of light is slightly larger than the field to be viewed.

4.5.11 Remove the eyepiece and view the back lens of the objective. Close the substage diaphragm until its diameter is just less than (2/3 - 4/5) the diameter of the back lens of the objective.

4.5.12 Replace the eyepiece, making sure that it is properly seated.

4.5.13 Adjust the general level of intensity of the illumination by closing or opening the field diaphragm. Do not change the setting of the substage diaphragm.

4.5.14 Illumination is now critical, and measurements of diameter may be made.

- Note 4: Unless great care is taken in obtaining critical illumination, the observed diameter of the fiber will be significantly changed by slight changes in the setting of the substage diaphragm.
- Note 5: If it is found that the substage condenser cannot be moved up high enough to provide critical illumination, remove the microscope slide and lay the specimen directly on the microscope stage.

4.6 Measure the diameter of the fiber at least three times along its length. Do not change the focus during any one measurement. Be very careful that there is no vibration present which may change the position of the fiber in the field during the course of a measurement.

## 5. CALIBRATION OF THE EYEPIECE

5.1 V<sup>(n)</sup> out changing the magnification of the microscope, remove the microscope slide carrying the fiber and replace it with a slide carrying a stage micrometer scale. This is the scale engraved on glass whose small divisions are 0.01 mm.</sup>

5.2 Bring the stage micrometer stage into sharp focus.

5.3 Determine the change in reading on the micrometer drum corresponding to a measured displacement of the image on stage micrometer scale.

5.3.1 In the case of the Filar micrometer eyepiece, set the hairline on any scale division and read its position on the eyepiece scale micrometer. Displace the hairline by rotating the eyepiece drum until it rests on another division of the micrometer scale a distance of at least 0.2 mm on the first position. Again read the position of the hairline on the eyepiece scale. Subtract the two eyepiece readings to obtain the relationship between the eyepiece scale reading and actual displacement on the micrometer scale (See 5.3.3).

5.3.2 In the case of the image-splitting eycpiece, read the eycpiece scale when the two images of the stage micrometer scale coincide. Displace one image relative to the other, by rotation of the eycpiece, through a distance of not less than 0.2 mm. Again read the eycpiece scale. Relate the difference between these two readings to the displacement of the image of the stage micrometer scale (See 5.3.3).

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# 5.3.3 Calibration factor = evepiece scale divisions

Example: for a displacement of 0.30 mm (300 microns) a reading on the eyepiece scale of 256 was obtained. The calibration factor for the eyepiece scale is then given by  $300 \div 256 = 1.17$  microns per division.

#### 6. CALCULATION

6.1 Convert all eyepiece scale readings to microns by using the conversion factor obtained as instructed in Section 5.

6.2 Determine the average diameter in microns for each fiber measured.

6.3 Calculate the average cross-sectional area for each fiber as follows (See Appendix VII):

$$A = KD^2$$

where D is the fiber diameter in microns

K = 0.7854 for the area in square microns

=  $1.217 \times 10^{-3}$  for area in square mils.

Note 6: This calculation assumes that the cross-section of the fiber is perfectly circular.

7. REPORT

For each fiber report

7.1 The number of fibers measured, and the number of measurements made on each fiber.

7.2 The average diameter for each fiber, and the range of measurement of diameter for all fibers in mils.

7.3 The standard deviation of individual measurements of diameter, if desired.

7.4 The average cross-sectional area in square mils.

B. CROSS-SECTIONING TECHNIQUE

#### 8. SCOPE

The cross-sectional area of elliptical or irregularly shaped fibers, such as graphitized viscose, cannot be determined by the techniques described above. Such fibers are embedded in a plastic, and standard metallurgical techniques are used to obtain an enlarged photograph of the cross-section, which can then be measured by using a planimeter.

#### 9. PROCEDURE

9.1 Support the fiber or a tuft of fibers in a vertical position, such that the free lower ends hang centrally within a metal ring of inside diameter approximately one inch, and height approximately 1-1/4 inch. Spray the inside of the ring with a release agent such as Teflon or a silicone, and support it on a surface coated with the release agent.

9.2 Mix suitable proportions of AB plastic, Castolite, Buehler Catalog No. 20-8120, and AB hardener, Castolite, Buehler Catalog No. 20-8122, according to the instructions supplied with the resin. Allow the resin to set 4 or 5 minutes (solution will change in color from green to clear) before carefully pouring the resin into the metal ring to a depth of at least one inch. Allow the mixture to set until gelled, then put the assembly into an oven at  $60^{\circ}$ C for 20 to 30 minutes. Return to room temperature and remove from mold.

9.3 Polish one surface of the resin plug containing the fiber using standard metallographic techniques. A suitable sequence of operations is as follows:

9.3.1 Use AB Supermet Silicon Carbide Grinding Paper of 180, 120 and 60 grit (Buehler Catalog No. 15-5112) for coarse grinding.

9.3.2 Continue using a polishing wheel covered with AB Kitten-Ear wool broadcloth using AB Micro Polish (5 micron alumina) followed by AB Alpha Micro Polish (0.3 micron alumina).

9.3.3 Carry out final polishing on a Syntron lapping polishing machine, first using Superme. polisher and then Polimet polisher. Depending upon the fiber involved, select a suitable combination of polishing materials from AB Polishing Alumina, AB Alpha Micro Polish, AB Gamma Micro Polish, and AB Gamma Polishing Alumina.

9.4 Such polishing methods are suitable for obtaining the cross-section of polymeric materials as well as of carbon or glass. Harder materials, such as boron, silicon carbide, etc., must be embedded in a metallic matrix having a hardness closer to the hardness of the fibrous material. Suitable embedding materials will have to be determined by experiment for each particular type of fibrous material.

9.5 Photograph the cross-section using a magnification which will give an approximate width of the cross-section of the fiber on the print of not less than 1 inch. A suitable instrument for obtaining such a photograph is the Bausch & Lomb Research Metallograph A1000. For most purposes, a 41X Achromatic objective used with a 15X eyepiece giving a final magnification on the print of about 2600X has been found to be satisfactory. Determine the exact magnification used by photographing a stage micrometer under the same conditions as were used for the fiber cross-section.

Note: If the width of the cross-section cannot be made as large as 1 inch, a negative will have to be obtained so that an enlarged print can be made. Otherwise the error in the measured cross-sectional area will be too large.

9.6 Measure the area of the fiber cross-section on the photographic print using a planimeter which can be read to 0.01 square inch or equivalent.

9.7 The cross-sectional area of the fiber is equal to the area of the projected image as determined from the planimeter divided by the square of the linear magnification.

#### C. DERIVED FROM LINEAR DENSITY

#### 10. PROCEDURE

10.1 Determine the linear density of the fiber as described in Method FMT-5.

10.2 Determine the specific gravity of the fiber as described in Method FMT-7.

10.3 Calculate the cross-sectional area as follows:

$$A = \frac{\text{Denier}}{9\rho \times 10^5} \text{ cm}^2$$
$$= \frac{\text{Denier}}{58.1 \rho \times 10^5} \text{ in}^2$$

where  $\rho$  = density in g/cc.

10.4 If the fiber cross-section is perfectly circular, the fiber diameter may be calculated as follows:

$$d = 1.13 \sqrt{A}$$

if A is in cm<sup>2</sup>, d is in cm. To get d in microns, multiply by  $10^4$  if A is in in<sup>2</sup>, d is in inches. To get d in mils, multiply by  $10^3$ 

# 11. EXAMPLE

if Denier = 3.5

$$\rho = 2.49 \text{ g/cm}^3$$

then 
$$A = \frac{3.5}{9 \times 2.49 \times 10^5} = 1.6 \times 10^{-6} \text{ cm}^2$$
  
or  $A = \frac{3.5}{58.1 \times 2.49 \times 10^5} = 2.4 \times 10^{-7} \text{ inch}^2$ 

For a fiber having a circular cross-section

d = 
$$1.13 \sqrt{1.6 \times 10^{-6}} = 1.13 (1.27 \times 10^{-3})$$
  
=  $1.4 \times 10^{-3} \text{ cm}$   
=  $14 \text{ microns}$   
or d =  $1.12 \sqrt{24 \times 10^{-8}} = 1.13 (4.9 \times 10^{-4})$   
=  $5.5 \times 10^{-4} \text{ in.}$   
=  $0.55 \text{ mils}$ 

#### METHOD FMT-7

## MEASUREMENT OF DENSITY OF FIBERS

## 1. INTRODUCTION

Three methods of measuring the specific gravity of fibers are described herein.

## A. DIRECT WEIGHING TECHNIQUE

#### 2. SCOPE

The density of fibers having a known and uniform cross-section may be determined by direct weighing of a measured length of fiber.

#### 3. BALANCE

Any balance having a precision of 2 to 3 micrograms can be used. The microbalance made by Mettler is suitable for this measurement.

#### 4. PROCEDURE

Cut an accurately measured length of fiber sufficient to give a total fiber weight of not less than 500 micrograms.

## 5. CALCULATION OF DENSITY

The density  $\rho = 127 \frac{w}{d^2 \ell}$  where w = weight in micrograms of a fiber having a length centimeters and a diameter d mils. Express the density to two decimal places.

#### 6. EXAMPLE

#### if

 $\ell$  = 1000 cm, d = 14 $\mu$ , and w = 3844 micrograms,

then

$$\rho = \frac{127 \times 3844}{14^2 \times 10^3} = 2.49 \text{ g/cm}^3$$

## B. DENSITY GRADIENT COLUMN

## 7. SCOPE

When the cross-sectional area of the fiber is not uniform or is unknown, the density may be determined directly by immersing segments of fiber in a suitably prepared density gradient column.

#### 8. PROCEDURE

8.1 The preparation and use of a density gradient column is described in detail in ASTM Method D1505-63T. This method also includes a table of suitable liquids which can be used to cover the density range from 1.0 to nearly 3.0.

8.2 Most of the fibers which are of interest to the Fibrous Materials Branch have a specific gravity which lies between 1 and 3.5. For most of these materials suitable liquids have been found to be orthodichlorobenzene and tetrabromoethane. Mixtures of these liquids cover the specific gravity range of 1.29 to 2.94. The sensitivity of the density gradient column should be adjusted to give a specific gravity accurate to the second decimal place.

8.3 Accurate temperature control is most important. Measurements must all be made with the density gradient column immersed in a water bath at  $70^{\circ} \pm 0.2^{\circ}$ F.

#### C. WESTPHAL BALANCE

#### 9. SCOPE

An alternative to the use of the density gradient column is a technique which employs the Westphal Balance (See Figure 16). The balance gives a direct reading of specific gravity when a calibrated plummet is weighed when immersed in a liquid having a specific gravity equal to that of the tiber.

#### 10. PROCEDURE

10.1 Fill a buret with orthodichlorobenzene (specific gravity 1.29) and another with tetrabromoethane (specific gravity 2.94), or with two alternative liquids covering a suitable specific gravity range. (See ASTM Method D1505-63T.)



Figure 16. Westphal Balance

10.2 Place a few short segments of fiber in a beaker immersed in a water bath controlled at  $70^{\circ} \pm 0.02^{\circ}$ F. Run in some liquid from each of the two burets. Stir to mix the two liquids thoroughly, and observe whether the fibers float or sink in the mixture. (Note: Accurate temperature control is essential to prevent convection currents from invalidating this observation.) If they float, slowly add more of the liquid having the lower specific gravity with constant stirring until the fibers appear to remain suspended in the liquid mixture. If the fibers sink, adjust the specific gravity of the mixture by adding some of the liquid of higher specific gravity. When the mixture appears to have the same specific gravity as the fibers, make final adjustments by running in no more than a few drops of liquid, stir, and allow to sit still for at least one minute before deciding whether the fiber is sinking or floating.

10.3 Read from the burets the amount of the two liquids which were mixed to give a mixture of the correct specific gravity. Make up sufficient volume of this mixture using the same proportions of the two liquids, to fill the cylinder of the Westphal balance. Check to see that the specific gravity of this large volume of mixed liquids is correct by dropping in a few segments of the measured fiber to see if it remains at equilibrium.

10.4 Hang the calibrated plummet on the arm of the Westphal balance and bring up the cylinder containing the liquid of specific gravity equal to that of the fiber so that the plummet is completely immersed. Adjust the balance weights so that the pointer on the end of the arm lies at the center of the scale. The specific gravity of the liquid can then be read directly off the balance.

10.5 The specific gravity of the fiber is equal to the specific gravity of the liquid. Report this to the second decimal place.

## D. ARCHIMEDES METHOD

1. BASIC METHOD

The sample is weighed in air and in a liquid of density lower than the sample, that will thoroughly wet the sample. Difference is the bouyant force, which is converted to sample volume by dividing by liquid density. Then weight in air divided by volume equals sample density.

#### 2. PRACTICE

The immersed sample weight is measured by hanging a coil of yarn or fiber on a hook, so that it is suspended freely in the container of liquid, which rests on a bridge over the balance pan. The hook is attached to the upper pan hook. Immersed weight of hook alone also must be obtained.

3. SET-UP





- 4. CALCULATIONS
  - If A = weight of sample in air, gms
    - H = weight of hook hanging in liquid, gms
    - F = weight of hook and sample in liquid, gms
  - $\rho_{\rm L}$  = density of liquid, gm/cm<sup>3</sup>
  - $\rho_{\rm S}$  = sample density, gm/cm<sup>3</sup>

Then: 
$$\rho_{\rm S} = \frac{A \rho_{\rm L}}{A + H - F}$$

## 5. LIMITATIONS, ADVANTAGES, DISADVANTAGES

5.1 Liquid must penetrate and wet sample thoroughly. Methanol works well for graphite.

5.2 Liquid must not be a solvent for sample.

5.3 Relatively large sample required. Thornel samples of 30 mgm handled easily; lower limit probably around 10 mgm.

5.4 Method is rapid and reproducible.

5.5 For porous fibers, result will depend on degree of liquid penetration into sample. Therefore, may 'e time-dependent.

5.6 With extreme care, may be adaptable to microbalance and samples under one (1) milligram.

## METHOD FMT-8

## MEASUREMENT OF INITIAL MODULUS FROM THE LOAD-ELONGATION CURVE

#### 1. INTRODUCTION

1.1 The initial modulus may be obtained from the slope of the tangent to the initial linear portion of the load-elongation curve, provided the elongation can be determined with sufficient accuracy. For this purpose, curves obtained as outlined in Method FMT-3 using the optical Strain Analyzer are the most reliable. However, for materials which can be tested in the Instron, results can also be obtained when a 10-inch gage length is used.

1.2 The significance of the value obtained by this technique will depend upon whether the curve shows an initial linear portion of appreciable length or not. If the load-elongation curve shows no initial linear portion, but curves continuously from the point of deviation from the zero-load axis, an initial slope may be drawn and a modulus calculated, but its practical significance in terms of the behavior of the material is questionable.

2. TEST CONDITIONS

2.1 Test all specimens in the optical strain analyzer as described in Method FMT-3.

2.2 Mount all specimens for Instron test using a 10-inch gage length, by one of the following methods:

2.2.1 Direct grip jaws, as outlined in Method FMT-1, Section 4.1.

2.2.2 Paper tab mounting, as outlined in Method FMT-14, Section 2.2

2.2.3 Epoxy tab, as outlined in Method FMT-1, Section 4.3.

2.3 Set the speed of operation of the instron as follows:

Yield Strain	Crosshead Speed	Chart Speed	
< 1%	0.2"/min.	20"/min.	
1-2%	0.2"/min.	10''/min.	
> 2%	0.2"/min.	2"/min.	

Note 1: If a specimen length sufficient to provide for a 10-inch gage length cannot be obtained, it may be possible to use a shorter gage length. However, this can influence the reliability of the measured modulus, particularly in the case of materials having a low yield strain, and a gage length of less than five inches should be used only with caution. In order to determine whether any desired gage length is long enough to give a reliable result, measure the modulus using several different gage lengths and plot modulus against gage length. The curve will indicate that below some value of gage length the modulus will be variable, while above this gage length it will remain essentially constant, or will vary in a unidirectional, consistent manner. Only gage lengths in the range which gives constant or consistently variable values of modulus should be used (See also Note 2.). If only very short gage lengths can be obtained, a better estimate of the tensile modulus may be obtained by measuring the bending modulus as described in Method FMT-10.

#### 3. PROCEDURE

Clamp the specimen in the Instron and operate the tester to obtain a load-elongation curve. Stop the crosshead as soon as the yield strain has been exceeded; it is unnecessary to run the curve up to rupture.

#### 4. RESULTS

- Note 2: Most high modulus fibers, if properly mounted, will give a load-elongation curve which has a clearly defined initial linear region, and the tangent whose slope defines the initial modulus can be drawn easily. In cases where the fiber is not completely straight initially, due to the presence of crimp or slack, the plot will have an initial section and will be concave upward. This part of the curve should be disregarded and the tangent drawn to the subsequent section which will rise more steeply and usually has an appreciable length which is more or less straight (See Figure 17).
- 4.1 Draw a tangent, SP, to the initial part of the load-elongation curve.
- 4.2 P is any point on this tangent such that the distance, SU, is at least 1 chart inch.
- Note 3. It is usually convenient to select P in such a way that the distance, SU, is a simple length on the chart, e.g., 2 inches, 5 inches, etc. (See Section 8).

4.3 Calculate the slope as

$$\Delta = \frac{OJ}{SU} \text{ (grams or pounds)}$$

4.4 Calculate the initial modulus as

$$E = \frac{MG}{A} \cdot \Delta$$

$$M = \frac{\text{chart speed}}{\text{speed of the pulling jaw}}$$

G = initial gage length

A = cross-sectional area

# 5. DETERMINATION OF TRUE MODULUS

Whenever the yield strain of the material being measured is below 1%, a check should be made to determine whether the observed modulus differs significantly from the true modulus. A method for doing this is as follows:

5.1 Initial Modulus Measurements

5.1.1 Errors in the measurement of initial modulus become larger as the magnitude of the modulus increases, or as the yield strain decreases. For example, in a material having 0.5% yield strain, the yield occurs in a 10-inch specimen at only 0.05 inches elongation. Displacement of the crosshead in an Instron tester cannot be measured with an accuracy of better than about ±0.001 inch, and sometimes the error may be larger than this. In addition, the

where



ELONGATION (CHART INCHES)

Figure 17. Determination of Initial Modulus

loading and load-measuring system in an Instron can deflect as much as 5-10 mils, or 10-20% of the elongation to be measured. Whenever low elongation materials are to be measured, therefore, it is important to determine whether the error due to deflection of the components of the tensile tester will have a significant influence on the results.

5.1.2 Tester deflections, or other factors such as jaw slippage which introduce similar errors, invariably increase with load. To a first approximation it is fair to assume that they are linearly dependent upon the applied load, and upon nothing else. Based upon this assumption, it is possible to carry out a simple experiment to determine the magnitude of these errors, and the extent to which the true modulus for the material differs from the observed modulus.

5.1.3 To accomplish this, measurements must be made on specimens of different gage length, for example, 1, 2.5, 5 and 10 inches. All specimens must be selected carefully to have the same cross-sectional area. If this cannot be done, the true modulus cannot be determined, or at best can only be determined approximately (perhaps no more accurately than the initially observed value).

## 5.2 Procedure

5.2.1 Obtain load-elongation curves for the specimens, adjusting the speed of operation of the machine to give the same strain rate for each gage length used.

Example	Gage Length (inches)	Crosshead Speed (inches/min)	
	1 2.5	0.02 0.05	
	5 10	0.1 0.2	

5.2.2 If the curves deviate abruptly from the zero-load axis and are linear right up to the yield, select a load on the linear portion of the curve just below the yield, and read the corresponding indicated elongation for each gage length. Convert these observations to strains by dividing by the corresponding gage length.

## Example

Gage Length (inches)	Elongation at Load P (inches)	Observed Strain	1 Gage Length (inch <sup>-1</sup> )
1	0.015	0.015	1.0
2.5	0.023	0.0092	0.40
5	0.035	0.0070	0.20
10	0.060	0.0060	0.10

5.2.3 Plot the observed strain (in absolute units, not percentage) against the reciprocal of the gage length. Draw a straight line through the points and extrapolate it back to the axis corresponding to a reciprocal gage length of 0. Read off the intercept on the strain axis in units of strain. In the example quoted in 2.2 this intercept is 0.0050.

#### 5.3 Calculation

5.3.1 Equate the value obtained for the intercept in absolute strain units to the quantity  $P/AE_{\rm T}$ , where

- P = the load at which the observed strain was determined for each gage length, in pounds
- A = the cross-sectional area of the specimens, in sq. in.

 $E_{T}$  = the true modulus, in psi.

5.3.2 In the example given, P was 0.50 lb, A was 24 sq. mils

Therefore 
$$0.0050 = \frac{0.50}{24 \times 10^{-6} \times E_{T}}$$

or

$$E_{T} = \frac{0.50}{24 \times 10^{-6} \times 0.0050} = 4.2 \times 10^{6} \text{ psi}$$

5.4 Derivation of the Formula

5.4.1 Let L = initial length of specimen

 $\Delta L_{obs}$  = observed increase in length caused by P

- P = ioad close to but less than the yield load
- A = cross-sectional area of specimen
- K = error in observed elongation due to machine deflection, whose magnitude depends only on P
- $E_{T} = true modulus$
- $E_{obs} = observed modulus$
- $\epsilon_{obs}$  = observed strain

5.4.2 Then 
$$\triangle L_{obs} = \frac{P/A}{E_T} \cdot L + K$$

and

$$\epsilon_{\rm obs} = \frac{\Delta L_{\rm obs}}{L} = \frac{P}{AE_{\rm T}} + \frac{K}{L}$$

5.4.3 A plot of  $\epsilon_{obs}$  against 1/L for fixed values of P and A will give a straight line whose slope is K and whose intercept on the 1/L = 0 line is P/AE<sub>T</sub>. Since P and A are both constant and known, E<sub>T</sub> can be calculated.

5,5 Alternative Procedure

5.5.1 
$$E_{obs} = \frac{P/A}{\epsilon_{obs}} = \frac{P/A}{P/AE_{T} + K/L}$$
$$\frac{1}{E_{obs}} = \frac{P/AE_{T} + K/L}{P/A} = \frac{1}{E_{T}} + \frac{KA}{P} \cdot \frac{1}{L}$$

5.5.2 A plot of  $1/E_{obs}$  against 1/L would be a much less satisfactory way of obtaining a value of  $E_T$ , for the plot is not a linear one (see Equation 3) and reliable extrapolation to the 1/L = 0 line will not be easy. However, if such an extrapolation were done, the intercept would be  $E_T$ .

## 6. REPORT

Report the following:

- 6.1 Type of fiber
- 6.2 Cross-section of fiber
- 6.3 Number of fibers tested
- 6.4 Average initial modulus in psi and range of individual values
- 6.5 Standard deviation of individual values

#### 7. CALCULATION

Symbol	Quantity	Units	How Measured
EI	initial modulus	pounds/sq in. (psi) grams/denier (gpd)	Appendix I
Δ	initial slope in chart units	pounds/chart inch grams/chart inch	Appendix I
М	magnification factor	chart speed (in./min) speed of pulling jaw (in./min)	
G	initial gage length	inches	
A	cross-sectional area	square mils	FMT-6
D	linear density	denier	FMT-5
Calculate	From	Formula	
E <sub>r</sub> psi	<b>Δ</b> , <b>M</b> , G, A	$E_{I} = \frac{\Delta MG}{A} \times 10^{6}$ for $\Delta$ in lb/chart in	

$$= \frac{\Delta MG}{A} \times 2.205 \times 10^3 \text{ for } \Delta \text{ in g/chart in}$$

Calculate	From	Formula (Continued)
$\mathbf{E}_{\mathbf{I}} \mathbf{g} \mathbf{p} \mathbf{d}$	∆, M, G, D	$E_{I} = \frac{\Delta MG}{D}$ for in $\Delta g$ /chart in
		$=\frac{\Delta MG}{D} \times 454$ for $\Delta$ in lb/chart in

8. EXAMPLE

If	OJ SU		0.099 lbs 8.00 chart inches
then	Δ	=	$1.24 \times 10^{-2}$ lb/chart inches
or if	OJ SU		33.7 grams 6.00 chart inches
then	Δ	=	5.62 grams/chart inch

for jaw speed of 0.2 inches/min

and chart speed of 2 inches/min

$$M = \frac{2}{0.2} = 10$$

G = 10.0 inches

A = 0.24 square mils

$$D = 3.5$$
 denier

$$\begin{array}{ccc} \underline{Calculate} & \underline{From} & \underline{Results} \\ E_{I} psi & \Delta, M, G, A & E_{I} = \frac{1.24 \times 10^{-2} \times 10 \times 10 \times 10^{6}}{0.24} \\ & = 5.2 \times 10^{6} psi \\ & \text{or} \\ E_{I} = \frac{5.62 \times 10 \times 10 \times 2.205 \times 10^{3}}{0.24} \\ & = 5.2 \times 10^{6} psi \\ E_{I} = \frac{5.62 \times 10 \times 10}{3.5} = 1.6 \times 10^{2} \text{ gpd} \\ & \text{or} \\ E_{I} = \frac{1.24 \times 10^{-2} \times 10 \times 10 \times 454}{2.5} \end{array}$$

$$E_{I} = \frac{1.24 \times 10^{-2} \times 10 \times 10 \times 45}{3.5}$$
$$= 1.6 \times 10^{2} \text{ gpd}$$

## METHOD FMT-9

## MEASUREMENT OF SHEAR MODULUS USING A TORSION PENDULUM

## 1. INTRODUCTION

The shear modulus of a length of filament is determined by attaching a calibrated bob to its end, suspending the assembly vertically, and setting the bob in torsional oscillation. The shear modulus can then be determined by measuring the period of torsional oscillation, the moment of inertia of the bob, and the length and diameter of the filament.

#### 2. APPARATUS

2.1 Basically, the equipment consists of a mount from which to hang the fiber-torsion bob assembly, and a series of torsion bobs having a range of moments of inertia. (see Figure 18.)

2.2 Fourteen calibration bobs are available, identified by the letters A through N. (See Figure 19.) The moment of inertia of each of these bobs is given in Table I.



Figure 18. Torsiometer

**4**9



Figure 19. Calibrated Torsion Bobs

#### TABLE I

#### INERTIA BOB CHARACTERISTICS

Disc Identity	Moment of Inertia in-lb sec <sup>2</sup>	Disc Identity	Moment of Inertia in-lb sec <sup>2</sup>
Α	$46.79 \times 10^{-6}$	Н	$0.74 \times 10^{-6}$
В	$23.56 \times 10^{-6}$	I	$29.6 \times 10^{-6}$
С	$11.68 \times 10^{-6}$	J	$16.1 \times 10^{-6}$
D	$3.44 \times 10^{-6}$	К	$6.85 \times 10^{-6}$
Е	$11.29 \times 10^{-6}$	L	$1.68 \times 10^{-6}$
F	$2.94 \times 10^{-6}$	М	$0.851 \times 10^{-6}$
G	$1.93 \times 10^{-6}$	N	$0.0988 \times 10^{-6}$

2.3 Discs A through D are equipped with a coarse pin vise capable of clamping circular fibers or fiber mounts having diameters between 25 and 55 mils. Discs E through H are equipped with a finer pin vise capable of clamping circular fibers or fiber mounts having diameters less than 25 mils. Generally speaking, discs A through D will be used for coarse fibers having a relatively high torsional rigidity, while E through H will be used for finer, less rigid materials. The cylindrical bobs, I through N, are drilled centrally to accommodate fibers directly, and can be used for materials having a low torsional modulus, such as polymeric fibers.

## 3. CALIBRATION

3.1 No calibration is necessary when the moment of inertia of the bob being used is known, as is the case with the calibrated bobs supplied with the instruments. If it is desirable for some reason to use an uncalibrated bob, its moment of inertia may be obtained by either of two methods:

3.2 If the bob is a uniformly shaped homogeneous solid, it may be possible to calculate its moment of inertia from its dimensions. The simplest shape of bob is a right circular cylinder in which the fiber is attached either to the exact center of the circular face so that the fiber axis is coincident with the axis of the cylinder, or exactly midway along the length of the cylinder so that the fiber axis is perpendicular to the axis of the cylinder.

3.3 If  $M = mass of the cylinder = \frac{weight in lb}{g} lb in^{-1} sec^2$ 

 $g = acceleration due to gravity = 386 in./sec^2$ 

 $\mathbf{r}$  = radius of the cylinder in inches

L = length of the cylinder in inches

then

Moment of Inertia I =  $\frac{M r^2}{2}$ 

when the fiber is attached to the center of the circular face, or

Moment  
of Inertia I = 
$$M(\frac{r^2}{4} + \frac{L^2}{12})$$

when the fiber is attached to the midpoint of the length of the filament I is given in inch-lb  $\sec^2$ .

**3.4** If the moment of inertia of the bob cannot easily be calculated from its dimensions, it may be obtained by comparison with a calibrated bob. Attach the calibrated bob to a filament of sufficient length so that the period of torsional oscillation is about 1 to 2 seconds. Determine the period of oscillation to the nearest 0.01 second by timing 10 to 50 oscillations with a stop watch. Measure the length of the filament to the nearest 0.01 inch with the aid of a traveling telescope.

3.5 Remove the calibrated bob and attach in its place the bob of unknown moment of inertia. Using as close as possible the same length of filament as was used with the calibrated bob, determine the period of oscillation of the filament carrying the bob to be calibrated. If

 $L_1$  = length of the filament attached to the calibrated bob in inches,

 $T_1$  = period of the torsion pendulum using the calibrated bob,

 $L_0 =$  length of filament carrying the bob to be calibrated in inches,

 $T_{0}$  = period of the torsion pendulum using the uncalibrated bob,

 $I_1$  = moment of inertia of calibrated bob in in. -lb sec<sup>2</sup>,

 $I_2$  = moment of inertia of uncalibrated bob in in. -lb sec<sup>2</sup>,

then

$$I_2 = I_1 \frac{L_1 T_2^2}{L_2 T_1^2}$$

4. PROCEDURE

4.1 From the material for which shear modulus is desired, select a single filament having a length in the range 10 inches to 24 inches, a uniform diameter along its length, and no visible sign of surface defects.

4.2 Clamp filaments having diameters not less than 10 mils and which are not too brittle directly into the pin vise jaws. Finer filaments, or brittle materials, may have to be cemented into protective sleeves at each end particularly when pin vise jaws are used. The protective sleeve may be a roll pin or a length of hypodermic needle having a central hole preferably about twice, but no more than five times, the filament diameter. Select two such protective sleeves having a length 1 to 1-1/2 inches. Side them onto the test filament so that the ends of the filament protrude at least two inches. Lightly coat a one-inch length on each end of the filament with a suitable adhesive (Shell the serves back to a point where the filament is centrally located in the central hole of the sleeve, and the adhesive forms a sharp demarcation line between the uncoated filament and the sleeve exactly at a level of the edge

of the sleeve. Allow sufficient time for the adhesive to set thoroughly. When bobs I through N are used, the fiber can be cemented directly into the hole in the bob, using the technique described above for attaching the protective sleeve.

4.3 Select a torsion bob having a moment of inertia which will give a period of torsional oscillation of one to two seconds. If the shear modulus of the filament is known approximately, it may be possible to estimate the moment of inertia which will give such a period. If it is not possible to estimate this in advance, it may be necessary to try two or three bobs before the proper one is obtained.

4.4 Clamp the torsion bob onto one end of the filament, allowing about 1/4 inch of the protective sleeve to protrude from the vise jaw, if a sleeve is used.

4.5 Clamp the other end of the filament in the jaws of the opper rotatable pin vise attached to the top bracket of the Torsiometer. Again allow about 1/4 inch of the protective sleeve to protrude from the jaws.

4.6 Adjust the protractor base symmetrically so that it is about 1/2 inch below the disc of the torsion bob.

4.7 Measure the free length of the test filament to the nearest 0.01 inch by using a measuring telescope. <u>Note</u>: If the filament is mounted in protective sleeves, it may be possible to measure its length before attaching the torsion bob and hanging the assembly in the Torsiometer. However, this method of measuring the length is likely to be less accurate than a measurement made with a measuring telescope.

4.8 Set the bob in oscillation by rapidly rotating the top supporting pin vise through about 15° of arc. Then clamp the top support with the thumb screw and allow the bob to oscillate. <u>Note:</u> The mass should oscillate smoothly without sideways motion and the total rotational amplitude should not exceed about 45°.

4.9 With the aid of a stopwatch, determine the time for 50 complete oscillations of the bob (the period of one complete oscillation consists, for example, of the time taken from one extreme position of the rotating bob through to the opposite extreme position and back to the first one again). If the damping of the oscillation is too great to permit 50 complete oscillations, time may be taken for a smaller number but never less than ten.

Note 1: Measurements may be made at temperatures up to 300°F by enclosing the assembly in a suitable chamber. For higher temperatures than this, the torsion bobs supplied cannot be used and a different set would have to be manufactured out of suitable high-temperature-resistant material.

#### 5. RESULTS

5.1 Calculate the period of one oscillation as follows:

$$\tau = \frac{T}{n}$$

where T = time in seconds for n complete oscillations.

5.2 Record the moment of inertia of the torsion bob used; the length of the filament, the diameter of the filament.

6. REPORT

Report the following:

- 6.1 Number of filaments tested
- 6.2 Average shear modulus and range of values obtained
- 6.3 Standard deviation of the shear modulus of individual filaments, when required.
- 6.4 Type and diameter of filament tested

## 7. CALCULATIONS

Symbol	Quantity	Units	How Measured
G	shear modulus	pounds/sq in. (psi) grams/denier (gpd)	
I	moment of iner- tia of torsion bob	inlb $\sec^2$	Section 3
L	filament length	inches	
d	filament diameter	mils	FMT-6
ρ	filament density	g/cc	FMT-7
1	polar moment of inertia of filament	in <sup>4</sup>	$J = 9.82 x$ $10^{-14} d^{4}$ for circular cross-section*
τ	period of one oscillation	sec	

Calculate	From	Formula
G psi	<b>Ι, ℓ , τ , </b> J	$G = 39.5 \frac{I \ell}{\tau^2 J}$

G psi,  $\rho$ 

G gpd

G gpd = G psi x 
$$\frac{1}{\rho}$$
 x 7.81 x 10<sup>-5</sup>

8. EXAMPLE

If  $I = 5.07 \times 10^{-11}$  in. -lb sec<sup>2</sup>

 $\ell$  = 12.21 inches

<sup>\*</sup> Derivation of the appropriate formula for non-circular sections must be done by approximation. See a text on elasticity such as S. Timoshenko and G. H. McCullough "Elements of Strength of Materials".

$\tau = \frac{76.5}{50}$ seconds	= 1.53 seconds	
$J = 9.82 \times 10^{-14}$	$(0.55)^4 = 8.99 \times 1$	$0^{-15}$ inch <sup>4</sup>
$\rho = 2.49 \text{ gms/cm}^2$	2	
Calculate	From	Results
G psi	L, ℓ, τ, J	$G = \frac{39.5 \times 5.07 \times 10^{-11} \times 12.21}{1.53^2 \times 8.99 \times 10^{-15}}$
		= $1.16 \times 10^6$ psi
G gpd	Gpsi $ ho$	G = 11.6 x $10^4$ x $\frac{1}{2.49}$ x 7.81 x $10^{-5}$
		= 3.64 gpd

## METHOD FMT-10

# MEASUREMENT OF BENDING MODULUS AND FLEXURAL STRENGTH OF FIBERS OR YARNS USING THE FLEX JIG

#### 1. INTRODUCTION

This technique is suitable only for very stiff fibers. The fiber is supported horizontally in two rings, a measured distance apart, and the relationship between the deflection of the center point and load applied at the center point is determined. The bending modulus so obtained can be used as an estimate of the tensile modulus for fibers whose length is too short to permit the use of Method FMT-8.

#### 2. APPARATUS

A simple device for carrying out this test has been designed to be mounted in the Instron. It is shown in Figure 20.

## 3. PROCEDURE

3.1 Set the Instron crosshead speed at 0.2 "/min and the chart speed at 20"/min.



Figure 20. Flex Jig for Measuring Fiber Flexural Rigidity

3.2 Select a full-scale load and separation between the two supporting rings from the fixed jig such that the load-deflection curve departs from linearity at a load greater than onequarter of the full-scale load. Do not use a ring separation less than ten times the diameter of the fiber being tested.

3.3 Set the initial separation between the two supporting rings with the aid of calipers so that this separation is known to within  $\pm 0.01$ ". Check that the rings are equidistant from the central slot within  $\pm 0.01$ ".

3.4 Raise the crosshead so that the thin plate which is mounted in the lower jaw enters the slot between the two supporting rings of the fixed jig. Stop the crosshead when the hole in the thin plate overlaps the holes in the supporting rings.

3.5 Select a fiber long enough to be supported in the rings, and measure its diameter accurately to the nearest  $0.25 \mu$  (See FMT-6.)

3.6 Slide the fiber carefully through the three holes and allow it to rest on the two supporting rings.

3.7 Lower the crosshead and plot a curve between deflection and force up to the point where it deviates significantly from a linear deflection curve; or where the curve indicates excessive slippage in the two supporting rings (i.e., curve contains many sharp peaks).

3.8 At any point, P, on the linear section of the curve or extrapolated line, read the load, F, and the corresponding number of chart inches, X, from the point at which the line leaves the zero-load axis.

4. RESULTS

Draw a tangent to the initial linear portion of the curve. At any point on this tangent, read off the load corresponding to a given deflection, for example, 0.1 inch.

### 5. REPORT

Report the following:

- 5.1 Number of fibers tested.
- 5.2 Average bending modulus and range of values obtained.
- 5.3 Standard deviation of the bending modulus of individual filaments, if desired.
- 5.4 Type and diameter of fiber tested.

#### 6. CALCULATION

Symbols	Quantity	Units	Measured
<sup>Е</sup> b	bending modulus	pounds/sq in. (psi) grams/denier (gpd)	
S <sub>b</sub>	flexural strength	pounds/sq in. (psi) grams/denier (gpd)	
l	distance between supports	inches	1000 TOT 400

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Symbols	Quantity	Units	How Measured
d	diameter of fiber	mil	FMT-6
ρ	density of fiber	g/cc	FMT-7
х	displacement of point	P chart inches	read from chart (See 3.8)
F	force corresponding t point P	o pounds	read from chart (See 3.8)
у	deflection for which for is read	orce inches	y = X/M
f	force at break	pounds	read from chart
Calculate	From	Formula	
E <sub>b</sub> psi	<i>l</i> , d, y, F	$E_{b} = 4.25 \times 10^{11} \frac{F\ell^{3}}{yd^{4}} \text{ for fiber}$ cross-section*	of circular
S <sub>b</sub> psi	<i>l</i> , d, f	$S_{b} = \frac{8f \ell}{\pi d^3} \times 10^9$	
E <sub>b</sub> gpd	E <sub>b</sub> psi	$E_{b}$ gpd = $E_{b}$ psi x $\frac{1}{\rho}$ x 7.81 x 10 <sup>-5</sup>	
S <sub>b</sub> gpd	S <sub>b</sub> psi	$S_{b} gpd = S_{b} psi x \frac{1}{\rho} x 7.81 x 10^{-5}$	

7. EXAMPLE

If  $\ell = 0.20$  inches

d = 4.84 mil (circular cross-section)

X = 4.7 inches

\* Derivation of the appropriate formula for non-circular sections must be done by approximation of the moment of inertia, using the expression

$$E \equiv \frac{F\ell^3}{48 \text{ yI}}, \qquad S \equiv \frac{8f\ell}{\pi d^3}$$

See a text on elasticity such as S. Timoshenko and G. H. McCullough "Elements of Strength of Materials".

$$M = \frac{20}{0.2} = 100$$
  
y =  $\frac{4.7}{100} = 0.047$  inches  
F = 0.423 lb  
 $\rho = 2.49 \text{ g/cm}^3$   
f = 0.141 lbs.

Calculate	From	Results
E <sub>b</sub> psi	l, d, y, F	$E_{b} = 4.25 \times 10^{11} \times \frac{0.423 \times 0.20^{3}}{0.047 \times 4.84^{4}} = 56 \times 10^{6} \text{ psi}$
E <sub>b</sub> gpd	E <sub>b</sub> psi,ρ	$E_{b} = \frac{56 \times 10^{6} \times 7.81 \times 10^{-5}}{2.49} = 1.8 \times 10^{3} \text{ gpd}$
S <sub>b</sub> psi	<b>l</b> , d, f	$S_{b} = \frac{8 \times 0.141 \times 0.20}{\pi \times 4.83^{3}} \times 10^{9} = 492 \times 10^{3} \text{ psi}$
s <sub>b</sub>	S <sub>b</sub> psi, ρ	$S_{b} = \frac{492 \times 10^{3} \times 7.81 \times 10^{-5}}{2.49} = 15.4 \text{ gpd}$

## METHOD FMT-11

## MEASUREMENT OF MODULUS OF A FIBER BY THE VIBRATING REED TECHNIQUE

## 1. INTRODUCTION

In this technique, modulus is measured by a dynamic method in which the resonant frequency of vibration of a fiber is determined. This dynamic modulus will not generally be the same, as the static modulus measured as described in FMT-10.

#### 2. APPARATUS

Suitable equipment for exciting the fiber is an Astatic crystal recording head X26 connected to a Hewlett-Packard Model 200AB audio oscillator having a frequency range of 20-40,000 cycles per second. (See Figure 21.)





#### 3. PROCEDURE

3.1 Select a fiber about 1-1/2" long. Insert the end of this fiber into a roll pin so that approximately 1" of free length protrudes. Measure this length accurately to the nearest 0.01". Measure the fiber diameter to the nearest 0.01 mil or  $0.25\mu$ . Insert the roll pin in the recording head.

3.2 Turn on the audio oscillator and slowly increase the frequency until the end of the fiber is seen to vibrate at maximum amplitude. Read this frequency, which is fundamental vibration of the fiber.

#### 4. CALCULATIONS

Symbol	Meaning	Units	How Measured
E <sub>b</sub>	modulus	pounds/square inch (psi) grams/denier (gpd)	
ł	fiber length	inches	
d	fiber diameter	mil	FMT-6
ρ	fiber density	g/cc	FMT-7
f	resonant fre- quency of fundamental	cps	
Calculate	From	Formula	
E <sub>b</sub> psi	l, f,p,d	$E_{b} = 1.15 \times 10^{-2} \times \frac{\ell^{4} f^{2} \rho}{d^{2}} \text{ for }$ cross-section*	circular
E <sub>b</sub> gpd	E <sub>b</sub> psi E <sub>b</sub>	$gpd = E_b psi x \frac{1}{\rho} x 7.81 x 10^{-5}$	

5. EXAMPLE

If

 $\ell$  = 0.97 inches f = 7.53 x 10<sup>3</sup> cps ho = 2.49 g/cm<sup>3</sup>

d = 0.55 mil (circular cross-section)

 $\begin{array}{c|c} \underline{\text{Calculate}} & \underline{\text{From}} & \underline{\text{Results}} \\ E_{\text{b}} \text{ psi} & \ell, \text{ f,} \rho, \text{ d} & E_{\text{b}} = \frac{1.15 \times 10^{-2} \text{ x } (0.97)^4 \text{ x } (7.53 \times 10^3)^2 \text{ x } 2.49)}{(0.55)^2} \\ &= 4.7 \times 10^6 \text{ psi} \\ \hline E_{\text{b}} \text{ gpd} & E_{\text{b}} \text{ psi,} \rho & E_{\text{b}} = \frac{4.7 \times 10^6 \text{ x } 7.81 \times 10^{-5}}{2.49} = 1.5 \times 10^2 \text{ gpd} \end{array}$ 

<sup>\*</sup> Derivation of the appropriate formula for non-circular sections must be done by approximamation of the moment of inertia. See references.

## 6. REFERENCES

Ballou and Smith, J. Applied Physics <u>20</u>, 493 (1949). Kärrholm and Schröder, Textile Research Journal <u>23</u>, 207 (1953). Machlan, G. R., WADC TR-55-290, 31 July 1955.

#### METHOD FMT-12

## MEASUREMENT OF BENDING MODULUS OF FIBERS USING SEARLE'S DOUBLE PENDULUM

#### 1. INTRODUCTION

In this technique the fiber whose bending modulus is desired is cemented as a cross-link between the bobs of two identical torsio<sup>-</sup> pendulums. The two pendulums are set into torsional oscillation in such a way that the fiber is continually bent, first in one direction and then in the opposite direction. The flexural rigidity of the fiber can be derived from measurement of the period of this complex vibrating system compared to the period of the individual torsion pendulums without the fiber attached as a cross-link.

#### 2. APPARATUS

Apparatus suitable for carrying out such measurements has been constructed for the Air Force Material Laboratory and a range of torsion bobs has been supplied which permits measurements to be made on fibers ranging from 1-1/2 denier polymerics to 5 mil boron (See Figure 22).



Figure 22. Searle Double Pendulum

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# 3. PROCEDURE

Instructions for using this equipment and the theory of its operation are given in detail in a report in the possession of the Fibrous Materials Branch, Air Force Materials Laboratory.

See also Owen, J. D. and Riding, G., Journal of Scientific Instruments 42, 14 (1965).

Owen, J. D., Journal of the Textile Institute 56, T329 (1965).

# METHOD FMT-13

# MEASUREMENT OF SONIC MODULUS

# 1. INTRODUCTION

The sonic modulus is measured by determining the wavelength of standing waves in the filament mounted under constant tension and driven at one end by a crystal recording head connected to an audio oscillator.

2. APPARATUS (See Figure 23).



Figure 23. Sonic Modulus Apparatus

- 2.1 Audio oscillator providing attenuated output over the range 100-2000 cps.
- 2.2 Crystal phonograph recording head.
- 2.3 Crystal phonograph playback cartridge.
- 2.4 Oscilloscope with both X and Y amplified inputs.
- 2.5 Rigid track with centimeter scale for measuring crystal separation.
- 3. PROCEDURE

3.1 Attach one end of the filament to the recording head needle by using sealing wax. Carry the filament over a pulley and attach the smallest weight to the other end which will hold the filament straight.

Note 1: For special purposes it may be desired to measure the effect of tension, or of strain, on the sonic modulus. While this is possible, it should be realized that if the tension exceeds the yield stress, the values which will be obtained are usually not reproducible because of specimen creep.

3.2 Bring the needle of the traversing crystal playback head into contact with the element at a distance of approximately 10 cm from the end which is attached to the recording head. Connect the output of this crystal to the Y-deflection terminals of the oscilloscope and the output of the audio oscillator to the X-deflection terminals.

3.3 Turn on the oscillator and set it to a frequency which is high enough to permit several readings of half wavelength to be made along the specimen. This is usually in the range of 10-15000 cps. Adjust the output attenuator and the X and Y axis gain of the oscilloscope so that the height and width of the figure obtained on the screen are approximately equal.

3.4 Traverse the receiving crystal along the filament and observe the change and shape of the pattern on the oscilloscope screen. This will change from an approximate circle to an ellipse with its major axis inclined somewhat away from the vertical, to a thinner ellipse, and eventually to a straight line inclined either to the left or the right. As the traversing continues the ellipse will again open up, slowly change into a circle, and again into an ellipse and a straight line inclined on the other side of vertical (See Figure 24). These figures represent changes in phase between the input and output signals. The distance along the filament corresponding to patterns which are exactly similar to one another corresponds to one wavelength of the standing waves which have been set up. Adjust the frequency in the audio oscillator so that this wave length lies in the range of 30 to 60 cm.

3.5 Move the traverse and crystal again back to a distance of about 10 cm from the recording head. Traverse slowly and steadily, stopping when the pattern on the oscilloscope screen is a straight line inclined either to the left or to the right of the vertical axis. Read the position of the traversing crystal at this point. Continue traversing the crystal until a straight line is obtained, inclined in the opposite direction. Again read the position of the crystal. This position is separated from the first position by one-half wave length. Continue the traversing, reading the position at which each straight line is obtained, until the full length of the filament has been traversed. If the signal is too weak when the distance between the recording head and the receiving crystal gets large, increase the output of the audio oscillator and make a corresponding adjustment in the X-axis gain of the oscilloscope in order to obtain an appropriate pattern.

**3.6** Determine the specific gravity of the filament by one of the techniques described in FMT-7.

4. CALCULATIONS

Symbol	Meaning	How Measured	
Es	sonic modulus	pounds/sq inch (psi) grams/denier (gpd)	
f	oscillator frequency	срв	
λ	wavelength	cm	
P	fiber density	g/cc	FMT7
Calculate	Fro	m	Formula
λ	$L_1, L_2, L_3$ - which a	are the successive	$\lambda = 2(L_2 - L_1)$
	readings obtained for traversing crystal w were seen on the osc	• the position of the hen straight lines illoscope screen	= $2(L_3 - L_2)$ , etc.



Figure 24. Waveforms for Sonic Modulus Determination

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Calculate	From	Formula
Es	<b>f</b> ,λ,ρ	$E_{\rm s} = 1.45 \times 10^{-5} {\rm f}^2 {\lambda}^2 \rho$
E <sub>s</sub> gpd	Es psi	$E_{g}$ gpd = $E_{g}$ psi $x \frac{1}{\rho} x 7.81 \times 10^{-5}$
5. EXAM	PLE	
If	$L_1 = 13.6 \text{ cm}$	
	$L_2 = 30.9 \text{ cm}$	
then	$\lambda = 2(32.7 - 13.6) = 38.2 \text{ cm}$	
and if	$\rho = 2.49 \text{ g/cm}^3$	
	$f = 1.08 \times 10^4 cps$	
Calculate	e From	Results

E <sub>s</sub> p	<b>8</b> i
------------------	------------

 $E_{s}$  gpd  $E_{s}$  psi, $\rho$ 

f,λ,ρ,

 $E_{s} = 1.45 \times 10^{-5} \times (1.08 \times 10^{4})^{2} (38.2)^{2}$  $x \ 2.49 = 6.14 \times 10^{6} \text{ psi}$  $E_{s} = \frac{6.14 \times 10^{6} \times 7.81 \times 10^{-5}}{2.49}$  $= 1.93 \times 10^{2} \text{ gpd}$ 

# METHOD FMT-14

# TENSILE TESTING OF YARNS USING THE INSTRON TENSILE TESTER

# 1. INTRODUCTION

The techniques used for the tensile testing of yarns are similar to those for testing fibers on the Instron tester, as described in Methods FMT-1 and -2, except that a ten-inch gage length is used and the method of mounting and speed of testing may be somewhat different, as described below.

# 2. SPECIMEN MOUNTING

#### 2.1 Direct Grip Jaws

2.1.1 Flat face jaws may be used for gripping yarns directly when there is no evidence of slippage or damage. Evidence of unsatisfactory gripping may be (1) slippage in the jaws, which is immediately visible on the chart trace (See Appendix III); (2) consistent breaking of the yarn adjacent to the edge of the jaw face, which is an indication that the yarn has been damaged by the clamping action; (3) an excessive amount of extension of the yarn between the faces of the jaws known as jaw penetration (See Appendix V); (4) breakage of very brittle yarns due to crushing while the jaws are being tightened. If any of these faults is detected, the jaw face may be modified or one of the other two methods of mounting can be used.

2.1.2 It is generally desirable to use standard Instron rubber-lined jaws. However, flat face steel jaws may be used in some cases, or may be modified by lining the jaws with masking tape which extends over the bottom of the jaw to cover the sharp edge.

#### 2.2 Paper Mounting

2.2.1 Cut a piece of brown Kraft paper 12 inches wide and at least 12 inches long. Draw parallel lines on the paper exactly 10 inches apart and approximately one inch from each edge.

2.2.2 Cut yarn specimens 12 inches long.

2.2.3 Hold a single yarn so that its end extends over the line drawn on the paper and up  $\iota_0$  the edge of the paper. Apply sealing wax over and around the end of the yarn beyond the line so that good adhesion is obtained to the paper.

2.2.4 Repeat this operation for the other end of the yarn, being careful to hold the yarn straight, but not stretched, and exactly at right angles to the two scribed lines.

2.2.5 Attach the required number of specimens of yarn side by side on the paper, leaving a space of at least one inch between adjacent specimens.

2.2.6 After the sealing wax has hardened, cut the paper carefully between the yarns so that each specimen is separately mounted on its own piece of paper.

2.2.7 This is now equivalent to the paper tab mounting used for fibers and should be handled in a similar way. After mounting in the jaws of the tensile tester, carefully cut the paper within about one inch of each jaw, leaving the yarn as the sole remaining connection between the two jaws of the tester.

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### 2.3 Epoxy Tab

An epoxy tab may be applied to each end of the yarn specimen in exactly the way described for fibers in Method FMT-1. It is important to handle the mounted yarn very carefully, holding an epoxy tab in each hand, for the tabs are relatively heavy and indiscriminate handling could damage the yarn prior to testing.

# 2.4 Capstan Jaws

2.4.1 When an accurate measure of elongation is not needed, Capstan jaws may be used. These are pictured in Figure 25. It is apparent that there is no clearly defined specimen gage length, since some elongation can take place over the snubbing surface around which the yarn is wrapped. An effective gage length can be determined (See Appendix V), but even this is unreliable for Capstan jaws. If a measure of tensile strength is the only thing needed, however, Capstan jaws are often convenient to use, and will hold the yarn effectively without damage in most cases.

2.4.2 It is important that the yarn be wrapped around the jaws exactly as shown in Figure 25. The yarn is first placed in the slot with the short end protruding from the side away from the axis of load application. Then it is wound around the movable segment, carried over the fixed segment, and then off the side nearest the load axis and across to the opposite jaw. There the winding is first around the fixed segment, then around the movable segment and through the slot in a direction away from the load axis. The yarn segment being tested must be coincident with the axis of load application, as indicated in figure 25. It will help, when tightening the jaws, to hold the moving segment tightly against the yarn with the hand, or with a lever when the jaws are so designed, as the yarn ends are pulled tight.

# 3. PROCEDURES

- 3.1 Set the crosshead speed at 0.5 inches/min, and the chart speed at 5 inches/min.
- 3.2 Carry out the test as described in Section 5 of Method FMT-2 for fibers.

#### 4. RESULTS

4.1 From the chart, determine the breaking load, the breaking elongation, and energy to rupture as described in Method FMT-1, and the initial modulus as described in Method FMT-8.

4.2 Determine the linear density of the yarn as described in Method FMT-5.

4.3 Determine the specific gravity of the fiber as described in Method FMT-7.

# 5. REPORT

Report the following:

- 5.1 Type of yarn tested
- 5.2 Denier of yarn and the number of fibers in a cross-section.
- 5.3 The type of mount used.
- 5.4 The tamber of yarn specimens tested.

5.5 The average breaking load in pounds to the nearest 0.1 lb and the range covered by the individual results.

- 5.6 The standard deviation of individual values of yarn breaking load, if desired.
- 5.7 The tensile strength in psi quoted to three significant figures.
- 5.8 The average breaking elongation and range of individual results to the nearest 0.01%.





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5.9 Standard deviation of breaking elongations of individual values, if desired.

5.10 The average energy to rupture, and the range of individual values obtained.

5.11 The standard deviation of energy to rupture of values for individual specimens, if desired.

5.12 The average initial modulus in psi and the range of values obtained.

5.13 The standard deviation of initial modulus values for individual specimens if desired.

# 6. CALCULATIONS

Symbols	Meaning	Units	How Measured
s <sub>T</sub>	tensile strength	pounds/square in (psi) grams/denier (gpd)	
$^{L}B$	breaking load	pounds grams	
D	yarn linear density	denier	FMT-5
ρ	fiber density	g/cc	FMT-7
Calculate	From	Formul	a
<sup>S</sup> T <sup>psi*</sup>	$L_{B}^{}, D, \rho$	$S_{T} = 5.81 \times 10^{6} \frac{L_{B} \rho}{D}$	for $L_B^{}$ in lb.
		= 1.28 x $10^4 \frac{L_B \rho}{D}$	for $L_B^{}$ in g.
$\mathbf{S}_{\mathbf{T}}^{}$ gpd	L <sub>B</sub> , D	$ST = \frac{L_B}{D} \times k$ where	$k = 1$ for $L_B$ in g.
			= 454 for $L_B$ in lb.
	psi <b>. p</b>	$S_{T} = psi x \frac{1}{x} 7.03 x$	× 10 <sup>-4</sup>
7. EXAMPLE			
		a <b>z</b> a za <sup>3</sup>	

If  $L_B = 5.69 \text{ lbs} = 2.58 \times 10^3 \text{ g}$ D = 309 denier = 1.43 g/cm<sup>3</sup>

<sup>\*</sup> For the calculation of tensile strength in psi, it is usually not possible to measure the yarn diameter in order to calculate the cross-sectional area because the fibers do not fill the whole of the cross-sectional area so calculated.

Calculate	From	Results
S <sub>T</sub> psi	L <sub>B</sub> , D, <i>p</i>	$S_{T} = \frac{5.81 \times 10^{6} \times 5.69 \times 1.43}{309}$
		= $1.53 \times 10^5$ psi
		or
		$S_{T} = \frac{1.28 \times 10^{4} \times 2.58 \times 10^{3} \times 1.43}{309}$
		= 1.53 x 10 <sup>5</sup> psi
$^{\rm S}{}_{\rm T}$ gpd	L <sub>B</sub> , D	$S_{T} = \frac{2.58 \times 10^{3}}{309} = 8.35 \text{ gpd}$
		or
		$S_{T} = \frac{453.6 \times 5.69}{309} = 8.35 \text{ gpd}$

# **METHOD FMT-15**

# TENSILE TESTING AT ELEVATED TEMPERATURES

Tensile tests can be carried out in the Instron tester up to temperatures of  $2000^{\circ}$ F by using either the CSI heated chamber or the clamshell oven. Because only direct grip mounting of the specimen can normally be used, these tests are more suitable for yarns than for single fibers.

# 1. SPECIFICATIONS

1.1 CSI Heated Chamber (Figure 26)

Temperature range	ambient to $1000^\circ \pm 2^\circ F$
Specimen length	Specimens up to 10 inches long can be used

1.2 Clamshell Oven (Figure 27) Temperature range Specimen length

up to 2000°F

Normally 3 or 3-1/2 inches but specimens up to 6-8 inches may be used









#### 2. OPERATION

2.1 Operating instructions are given in manuals available at the Fibrous Materials Branch, Air Force Materials Laboratory. Any of the test conditions called for in methods FMT-1, FMT-2, or FMT-14 can be used.

2.2 The use of paper or epoxy tabs for mounting the specimen is usually not possible because of the elevated temperatures involved. Specimens requiring such special mounting may not be suitable for this type of testing on the Instron. Alternative methods for such materials are described in FMT-3 and FMT-4.

 $\frac{\text{CAUTION:}}{\text{ing be worn when the door of the heated oven is open.}}$ 

Protective clothing should be used as follows:

(1) 300°F to 500°F - face mask, reflective gloves with liners, reflective chest and arm shield or protective coat.

(2)  $500^{\circ}$ F to  $1000^{\circ}$ F - as in (1) except wear reflective hood in place of face mask.

(3) 1000°F and above - as (2) except wear reflective coat.

## 3. RESULTS

3.1 Breaking loads can be read off the chart in the usual way. Tensile strengths cannot be calculated accurately unless the true cross-sectional area of the specimen at the temperature of the test is known or can be calculated from a knowledge of the room temperature cross-section and the temperature coefficient of expansion of the material.

3.2 While absolute extensions can be read for the chart, percent elongations can only be calculated accurately if the true length of the specimen at the elevated temperature is known or can be estimated.

3.3 In the CSI heated chamber, i.e., at temperatures below 1000°F, this may be measured directly. Insert a specimen in the jaws in the normal way and bring the oven up to temperature. After the desired dwell time prior to testing, manually adjust the crosshead in a downward position until a load is just visible on the chart. Open the door of the chamber and quickly measure the jaw separation with a rule. Take this to be the true gage length of all specimens of the same material which are tested at the temperature for which the corrected gage length was measured.

3.4 Direct measurement of the true gage length of a hot specimen cannot be made in the clamshell oven at temperatures over 1000°F. In this case, the gage length must be calculated from the room temperature length of the specimen and the expansion characteristics of the specimen material, if known.

3.5 If a more accurate measure of modulus or breaking elongation at elevated temperature is needed, the technique described in FMT-3 should be used.

#### 4. REPORT

4.1 In addition to the data required in the appropriate tensile test method describing room temperature tests, report the following.

# 4.1.1 Test Temperature

4.1.2 Dwell time at temperature prior to testing.

# METHOD FMT-16

# YARN FLEX TESTING

# 1. INTRODUCTION

In this equipment, the yarn is held under a fixed tension and bent repeatedly through a fixed angle over a rod until breakage occurs.

# 2. APPARATUS

The apparatus is pictured in Figure 28. One end of the specimen is attached to a rocking arm which bends the yarn around a 1/16-inch diameter rod between the angles of 20 and 100 degrees at a rate of 89 cycles/minute. The other end of the specimen is attached to a cylinder carrying lead shot, whose weight can be adjusted to provide the desired tension.

# 3. SPECIMEN MOUNTING

Cut each specimen about 4 inches long. Attach the yarn specimens to cardboard tabs as described in Method FMT-1, Section 4.2.

# 4. PROCEDURE

4.1 Fill the tensioning cylinders with lead shot to provide the desired tension, which is usually some simple fraction, for example, half, of the breaking strength of the yarn.

4.2 Clamp one end of each cardboard tab to the rocking arm and attach the other end to the hook on the top of the tensioning weight.

4.3 Cut both sides of the tab so that the yarn alone supports the tensioning weight (cf FMT-1, Paragraph 5.2.2).

4.4 Start the oscillation and permit the machine to run until all specimens have broken. The number of cycles required to break each specimen will be recorded in individual counters connected to each specimen position.

#### 5. REPORT

Report the following:

5.1 The type and denier of yarn used.

5.2 The number of specimens tested.

5.3 The average number of cycles to break and the range of values obtained.

5.4 The standard deviation of the individual values obtained, if desired.





Figure 28. Yarn Flex Tester

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#### APPENDIX I

# PARAMETERS DERIVED FROM THE LOAD-ELONGATION CURVE (See Figure 29.)

Load-Elongation Parameters

Magnification Ratio  $M = \frac{\text{Chart Speed}}{\text{Speed of pulling jaw}}$ 

True gage length  $\ell g$  = Initial jaw separation +  $\frac{OS}{M}$ 

Yield: The yield point is determined graphically as follows:

1. For materials having an obvious transition from elastic to flow-type deformations, draw tangents to the Hookean and yield portions of the load-elongation curve. The point where the bisector of the angle between these tangents meets the load-elongation curve is defined as the yield point.

2. For materials which do not have a welldefined yield, e. g., some metal fibers, use an offset method of construction to locate the yield point. Draw a tangent AB to the initial portion of the load-elongation curve. Draw a line CD parallel to AB, and passing through a point C on the zero-load axis equivalent to 0.2% extension. The yield point is defined by the intersection of this line with the loadelongation curve. (For additional information see ASTM Method E8-61T)



Elastic Limit: The point at which the Hookean portion of the load elongation curve deviates from a straight line, at the transition to the yield region.

Break: The breaking load is the maximum load encountered in a tensile test carried to rupture. The breaking extension is the extension at the breaking load, related to the true gage length.

$$L_B = \frac{SV}{M\ell g} \times 100\%$$

The work done in breaking the specimen, or the energy to break, is determined by measuring the area between the zero-load line and the load-elongation curve between the start of the test and the break.

Energy to break = Area RYBVR sq. in. 
$$x \frac{\text{full scale load}}{\text{chart width (inches)}^*} x \frac{1}{M}$$

\* The width of an Instron chart is 9.53 inches



L = Failure or Rupture Load



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This quantity is often divided by the specimen length to give energy to break per unit length of specimen.\*

Failure or Rupture: The failure or rupture point is that point on the load-elongation curve where the load falls abruptly to zero, indicating that the specimen has broken apart. This point is often the same as the break point. When it is not, it may happen that it is not sufficiently defined to be considered determinable.

Initial Modulus: The initial modulus is derived from the slope of the tangent SP to the initial part of the load-elongation curve.

P is any point on this tangent such that the distance SU is at least 1 chart inch.

The slope is given by

 $\Delta = \frac{OJ}{SU}$  (grams or pounds) (chart inches)

Initial modulus,  $E = \Delta \frac{MG}{A}$ 

Where G is the initial gage length And A is the cross-sectional area

Note: Modulus is often given in grams/unit linear density, e. g., g/tex or g/den. In this case  $E = \Delta \frac{MG}{D}$ , where D is the linear density.

<sup>\*</sup> Breaking elongation has been measured from the point S, that is, it does not include the elongation resulting from crimp removal. This is because such a measure is a basic property of the fibrous material, which is usually of prime interest. The service performance of the fiber or yarn may be more influenced, however, by the <u>total</u> elongation to break, obtained by adding the crimp elongation to the breaking elongation.

Energy to break has been measured from the point R, so that it includes the energy required to remove the crimp. This not consistent with the method of measuring breaking elongation, primarily because it can be obtained from an integrator reading, while the energy to break, excluding crimp removal, cannot. If the amount of energy required for crimp removal is desired, it may be obtained by measuring the area between the initial part of the curve and the tangent SP, using a planimeter.

# APPENDIX II

# DETERMINATION OF MAXIMUM SPEED OF RESPONSE OF THE RECORDER

1. Determine the maximum speed of response of the recorder as follows:

1.1 Calibrate the Instron in the normal way.

1.2 Set the FULL SCALE LOAD switch to a low range.

1.3 Select the chart speed to be used for testing, and start the chart paper running.

1.4 Quickly turn the COARSE BALANCE switch one or two stops in a clockwise direction. This is equivalent to applying a load quickly to the load cell.

1.5 The recorder pen will draw a straight line on the chart, whose slope represents the maximum possible rate of traverse of the recorder pen.

2. If any part of the load-elongation curve, obtained while testing a fiber or yarn, has more than one-half the slope of the line representing maximum speed of the pen response for the same chart speed, the trace must be considered unreliable. Repeat the test using a lower rate of specimen extension.

3. Lower rates of specimen extension may be obtained by reducing the crosshead speed or by increasing the specimen length for the same crosshead speed.

# APPENDIX III

# DETECTION OF JAW SLIPPAGE

When a specimen slips from the jaws of the tensile tester, the load-elongation curve which is obtained is incorrect and must be discarded. Occasionally the presence of jaw slippage can only be detected by careful observation of the specimen during the course of the test. Usually, however, certain characteristic patterns show up in the load-elongation curve which can be taken as definite evidence of jaw slippage. Some typical examples of these patterns are shown in Figure 20.

#### Curve A

This is the type of curve that might be obtained from a fiber which has been attached by adhesive to a tab. The sawtooth points which are present on the rising portion of the curve indicate slippage of the fiber in the adhesive. The presence of a sharp drop, followed by a further increase in load, can always be taken as sufficient reason for discarding the test.

# Curves B and C

Curves B and C are typical of the results obtained when slippage occurs for a fiber or a yarn which has been clamped directly into the jaws. When such slippage occurs, it usually continues, so that although the load may start to build up slightly after an abrupt drop as in Curve B, often it will remain constant or gradually fall off as the test proceeds, as in Curve C. Any sign of a smooth falling off of load may be taken as an indication of slippage and the test should be discarded. Falling off of load in a yarn test which is characterized by jagged peaks usually indicates successive breakage of individual filaments, and need not be a cause for rejecting the test results. (See, for example, Curve C, Figure 30.)



**ELONGATION** Figure 30. Effect of Jaw Slippage for Load-Elongation Diagram

#### APPENDIX IV

# MEASUREMENT OF ENERGY TO RUPTURE

The Instron integrator is usually the simplest way to obtain a measurement of the energy required to break a specimen. However, there are cases in which this integrator will not give the proper value, and in these cases, one of the alternative techniques outlined in Method FMT-2 must be used.

Figure 31 shows three load-elongation curves having somewhat different characteristics. Curve A was obtained from a single fiber and shows a sharp break with the load falling abruptly to zero. For this case, the Instron integrator will give a reliable value which would correspond exactly to the value obtained by using a planimeter, for example.

Curve B was obtained for a specimen which did not break abruptly. In this case, the integrator will give the area under the curve until the load has fallen to zero. The area which defines the energy to break the specimen is contained within the dashed line which has been drawn from the point of maximum load. It can be seen that the integrator reading, therefore, would be somewhat higher than the properly determined value, and in this case the integrator should not be used.

Curve C is an even more extreme example of the type illustrated by Curve B. This is the case of a yarn in which the break has not been abrupt, but rather the filaments have broken one by one, resulting in a characteristic jagged decay of load from the point of maximum load, which is by definition the breaking point, to zero load. In this case, the Instron integrator would give a value which is much too high. Again, the planimeter or cutting-and-weighing technique would have to be used in order to obtain the correct value of the energy to break.

In summary, the Instron integrator is a reliable instrument so long as the specimen breaks cleanly so that the load falls abruptly from its maximum value to zero. In any case where this does not occur, the integrator may not be used.\* Alternative methods have been described in FMT-2.

<sup>\*</sup> A fairly reliable value may still be obtained if the operator manually stops the integrator as soon as an indication of load drop is observed. This method, however, involves accurate judgment on the part of the operator, which may sometimes be faulty. It should only be used by experienced operators, and only for materials for which the load falls rather rapidly.





#### APPENDIX V

# JAW PENETRATION EFFECTS - DETERMINATION OF EFFECTIVE GAGE LENGTH

# 1. INTRODUCTION

1.1 If the pressure of the jaws on the clamped specimen is not uniform throughout the whole of the clamped length, a certain amount of specimen extension may take place within the jaws. When this happens, if the elongation is measured from the charts and a calculation of percent elongation is based on the separation between the jaws at the start of the test, the resulting figure will be in error because of the fact that the length of specimen which has actually extended is longer than its initial free length.

1.2 Extension of the specimen within the jaws is referred to as jaw penetration. It may not be apparent from examining the load-elongation curve whether jaw penetration has occurred or not, because it usually shows no indication of stick-slip, but rather the within-jaw extension occurs in proport<sup>i</sup> in to the applied load. As a result, the load-elongation curve is apt to remain smooth and apparently normal.

1.3 If there is any reason to suppose that jaw penetration effects may be influencing the results, a technique is available for determining the effective magnitude of these effects, and of calculating an effective gage length, which will be somewhat longer than the initial jaw separation, and which should be used as a basis for calculating percent elongation. This technique is based upon the fact that the slippage of the fiber within the clamped zone is a function of the applied force, but not of the gage length. Therefore, a plot of elongation against gage length at a given force may not give a line which passes through the origin, but may be displaced along the elongation axis. This provides a means of determining whether jaw penetration exists, and its magnitude.

1.4 A technique for determining an effective gage length is described in ASTM method D1906-62T. Its use in connection with high modulus materials is discussed below.

- Note 1: This estimate of effective gage length is based upon the assumption that if tests are carried out at two or more gage lengths, the only difference between these tests is the relationship between the magnitude of the absolute elongation of the specimen and the elongation of the specimen within the jaws. This presupposes that the fiber properties are uniform along the length of the fiber, and that the clamping can be done in a reproducible manner from test to test. If the fiber is known to be significantly non-uniform along its length, effective gage length cannot be determined reliably.
- Note 2: Because this technique requires that fibers or yarns be tested using more than one gage length, it cannot be used for fibers which have been mounted on the cardboard tab, which fixes the gage length at one inch. If necessary, however, attachment of longer lengths of fiber to the same cardboard material using the same sealing wax technique could be used for comparison.

# 2. PROCEDURE

2.1 Mount fiber specimens, using the technique to be investigated, having lengths of 1, 5, and 10 inches. At least five specimens at each gage length are needed.

2.2 Clamp the fibers in the usual way, being careful to tighten the clamps in exactly the same manner for each test.

2.3 Plot load-elongation curves to rupture for each specimen, using the following test conditions:

2.3.1 Chart speed 20 inches per minute

2.3.2	Crosshead speed for 1" gage length	0.2 "/min
	for 5" gage length	1.0 "/min
	for 10" gage length	<b>2.</b> 0 "/min

2.4 Read off these curves the absolute elongation for each of the gage lengths corresponding to selected values of load, for example, 20%, 40%, 60%, and 80% of the average breaking load, or some similar distribution. Average the values of elongation for each load and gage length.

2.5 Plot a family of lines showing the relationship between elongation and gage length for each load. This will give a set of curves similar to those shown in Figure 32.

Note 3: If these curves are not all essentially linear, examine the variability between results obtained from the five specimens tested at each gage length. If the range of values of elongation at any load is more than 25% of the mean, the between-specimen variability is too high for this technique to give a meaningful result, and the effective gage length is indeterminate.

2.6 Extrapolate the lines down to the zero-length axis. The intercepts so obtained are a measure of the length of the specimen which is being stretched within the jaws at each load. Add this quantity to the initial jaw separation to obtain a value for the effective gage length for each load considered.

2.7 If needed, plot a curve between effective gage length and load.

2.8 When calculating the percentage elongation at any load value, read off from this curve the effective gage length corresponding to the load, and use this as the basis for calculating the percentage extension:

% extension =  $\frac{\text{absolute extension}}{\text{effective gage length}} \times 100\%$ 





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# APPENDIX VI

# ESTIMATION OF STANDARD DEVIATION

1. For less than 20 observations.

If the number of observations is less than 20, the standard deviation may be estimated from the range of values contained in the observations:

if

n = number of observations

d = difference between highest and lowest observation

then

s, the standard deviation = kd

where k has the value given in the following table:

<u>n</u>	<u>_k</u>	<u>n</u>	ĸ	n	<u>_k</u>	<u>n</u>	k
1		6	0.3946	11	0.3152	16	0.2831
2	0.8862	7	0.3698	12	0.3069	17	0.2787
3	0.5908	8	0.3512	13	0.2998	18	0.2747
4	0.4857	9	0.3367	14	0.2935	19	0.2711
5	0.4299	10	0.3249	15	0.2880	20	0.2677

2. A more accurate method is based upon the definition of standard deviation

$$s = \sqrt{\frac{(x - \bar{x})^2}{n-1}}$$
 where x is any observation, and  $\bar{x}$  is the mean of n observations

This is algebraically identical to another expression more suited to machine calculation.

$$s = \sqrt{\frac{n\Sigma x^2 - (\Sigma x)^2}{n - 1}}$$

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0.0001 0.00008 1.05 1.10 0.8	C.00008 1.05 1.10 0.5	1.05 1.10 0.8	1.10 0.8	0.0	366	2.05	4.20	3.30	3.05	9.30	7.31	4.05	16.4	12.9
0.0004 0.0003 1.10 1.21 0.9	C.0003 1.10 1.21 0.9	1.10 1.21 0.9	1.21 0.9	0.9	50	2.10	4.41	3.46	3.10	9.61	7.55	4.10	16.8	13.2
0.0009 0.0007 1.15 1.32 1.04	0.0007 1.15 1.32 1.04	1.15 1.32 1.04	1.32 1.04	1.04	.+	2.15	4.62	3.63	3.15	9.92	7.79	4.15	17.2	13.5
0.0016 0.0012 1.20 1.44 1.13	0.0012 1.20 1.44 1.13	1.20 1.44 1.13	1.44 1.13	1.13	~	2.20	4.84	3.80	3.20	10.2	8.04	4.20	17.6	13.8
0.0025 C.0020 1.25 1.56 1.23	C.0020 1.25 1.56 1.23	1.25 1.56 1.23	1.56 1.23	1.23	_	2.25	5.06	3.98	3.25	10.6	8.30	4.25	18.1	14.2
0.010 0.0078 1.30 1.69 1.33	C.0078 1.30 1.69 1.33	1.30 1.69 1.33	1.69 1.33	1.33		2.30	5.29	4.15	3.30	10.9	8.55	4.30	18.5	14.5
0.022 0.018 1.35 1.82 1.43	0.018 1.35 1.82 1.43	1.35 1.82 1.43	1.82 1.43	1.43		2.35	5.52	4.34	3.35	11.2	8.81	4.35	18.9	14.9
0.040 C.03i 1.40 1.96 1.54	C.031 1.40 1.96 1.54	1.40 1.96 1.54	1.96 1.54	1.54		2.40	5.76	4.52	3.40	11.6	9.08	4.40	19.4	15.2
0.062 0.049 1.45 2.10 1.65	0.049 1.45 2.10 1.65	1.45 2.10 1.65	2.10 1.65	1.65		2.45	6.00	4.71	3.45	11.9	9.35	4.45	19.8	15.5
0.090 C.071 1.50 2.25 1.77	C.071 1.50 2.25 1.77	1.50 2.25 1.77	2.25 1.77	1.77		2.50	6.25	4.91	3.50	12.2	9.62	4.50	20.2	15.9
0.122 0.096 1.55 2.40 1.89	C.096 1.55 2.40 1.89	1.55 2.40 1.89	2.40 1.89	1.89		2.55	6.50	5.11	3.55	12.6	9.90	4.55	20.7	16.3
0.160 C.126 1.60 2.56 2.01	C.126 1.60 2.56 2.01	1.60 2.56 2.01	2.56 2.01	2.01		2.60	6.76	5.31	3.60	13.0	10.2	4.60	21.2	16.6
0.202 0.159 1.65 2.72 2.14	0.159 1.65 2.72 2.14	1.65 2.72 2.14	2.72 2.14	2.14		2.65	7.02	5.51	3.65	13.3	10.5	4.65	21.6	17.0
0.250 C.196 1.70 2.89 2.27	C.196 1.70 2.89 2.27	1.70 2.89 2.27	2.89 2.27	2.27		2.70	7.29	5.72	3.70	13.7	10.8	4.70	22.1	17.3
0.302 0.237 1.75 3.06 2.40	0.237 1.75 3.06 2.40	1.75 3.06 2.40	3.06 2.40	2.40		2.75	7.56	5.94	3.75	14.1	11.0	4.75	22.6	17.7
0.360 0.283 1.80 3.24 2.54	0.283 1.80 3.24 2.54	1.80 3.24 2.54	3.24 2.54	2.54		2.80	7.84	6.16	3.80	14.4	11.3	4.80	23.0	18.1
0.422 0.332 1.85 3.42 2.69	0.332 1.85 3.42 2.69	1.85 3.42 2.69	3.42 2.69	2.69		2.85	8.12	6.38	3.85	14.8	11.6	4.85	23.5	18.5
0.490 0.385 1.90 3.61 2.84	0.385 1.90 3.61 2.84	1.90 3.61 2.84	3.61 2.84	2.84		2.90	8.41	6.60	3.90	15.2	11.9	4.90	24.0	18.9
0.562 C.442 1.95 3.80 2.99	C.442 1.95 3.80 2.99	1.95 3.80 2.99	3.80 2.99	2.99		2.95	8.70	6.83	3.95	15.6	12.2	4.95	24.5	19.2
0.640 0.503 2.00 4.00 3.14	0.503 2.00 4.00 3.14	2.00 4.00 3.14	4.00 3.14	3.14		3.00	9.00	7.07	4.00	16.0	12.5	5.00	25.0	19.6
0.722 C.567	C.567						•		} 	)   	) ] 4	> > •	•	· · · ·
0.810 0.636	0.636													
0.902 C.709	C.709													
1.00 C.785	C.785													

# APPENDIX VII (Continued)

AREA	OF	A	CIRCLE	OF	DIAMETER	D	MILS
------	----	---	--------	----	----------	---	------

$A = \frac{\pi d^2}{4} = 0.7854 d^2 (\text{microns})^2 = 1.217 \times 10^{-3} d^2 (\text{mil})^2$									
d	d <sup>2</sup>	Area		d	d <sup>2</sup>	Area			
μ	$\mu^2$	$\mu^2$	mil <sup>2</sup>	μ	μ2	μ2	mi1 <sup>2</sup>		
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33	$ \begin{array}{c} 1\\ 4\\ 9\\ 16\\ 25\\ 36\\ 49\\ 64\\ 81\\ 100\\ 121\\ 144\\ 169\\ 196\\ 225\\ 256\\ 289\\ 324\\ 361\\ 400\\ 441\\ 484\\ 529\\ 576\\ 625\\ 676\\ 729\\ 784\\ 841\\ 900\\ 961\\ 1024\\ 1089 \end{array} $	0.78 3.1 7.1 12.6 19.6 28.3 38.5 50.3 63.6 78.5 95.0 113. 133. 154. 177. 201. 227. 254. 284. 314. 346. 380. 415. 452. 491. 530. 572. 616. 660. 707. 755. 804. 855	$\begin{array}{c} 0.0012\\ 0.0049\\ 0.011\\ 0.019\\ 0.030\\ 0.044\\ 0.060\\ 0.078\\ 0.099\\ 0.122\\ 0.147\\ 0.175\\ 0.206\\ 0.239\\ 0.274\\ 0.312\\ 0.352\\ 0.394\\ 0.440\\ 0.487\\ 0.536\\ 0.536\\ 0.589\\ 0.643\\ 0.536\\ 0.589\\ 0.643\\ 0.701\\ 0.761\\ 0.822\\ 0.887\\ 0.955\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.10\\ 1.17\\ 1.25\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1.02\\ 1$	$\begin{array}{c} 36\\ 37\\ 38\\ 39\\ 40\\ 41\\ 42\\ 43\\ 44\\ 45\\ 46\\ 47\\ 48\\ 49\\ 50\\ 51\\ 52\\ 53\\ 54\\ 55\\ 56\\ 57\\ 58\\ 59\\ 60\\ 61\\ 62\\ 63\\ 64\\ 65\\ 66\\ 67\\ 7\end{array}$	$\begin{array}{c} 1296\\ 1369\\ 1444\\ 1521\\ 1600\\ 1681\\ 1764\\ 1849\\ 1936\\ 2025\\ 2116\\ 2209\\ 2304\\ 2401\\ 2500\\ 2601\\ 2704\\ 2809\\ 2916\\ 3025\\ 3136\\ 3249\\ 3364\\ 3481\\ 3600\\ 3721\\ 3844\\ 3969\\ 4096\\ 4225\\ 4356\\ 4489\\ \end{array}$	1020. 1070. 1130. 1190. 1260. 1320. 1380. 1450. 1520. 1590. 1660. 1730. 1810. 1890. 1960. 2040. 2120. 2210. 2290. 2380. 2460. 2550. 2640. 2730. 2830. 2920. 3020. 3120. 3220. 3320. 3420. 3530.	m11 1.58 1.67 1.76 1.85 1.95 2.05 2.15 2.25 2.36 2.46 2.58 2.69 2.80 2.92 3.04 3.17 3.29 3.42 3.55 3.68 3.82 3.96 4.10 4.24 4.38 4.53 4.68 4.83 4.99 5.14 5.30 5.47		
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# APPENDIX VII (Continued)

# AREA OF A CIRCLE OF DIAMETER D MILS

d	d <sup>2</sup>	Area		d	2	Area	
μ	μ2	μ2	mil <sup>2</sup>	μ	μ2	μ2	mil <sup>2</sup>
71	5041	3960	6.14	106	11236	8820	13.7
72	5184	4070	6.31	107	11449	8990	13.9
73	5329	4180	6.49	108	11664	9160	14.2
74	5476	4300	6.67	109	11881	9330	14.5
75	5625	4420	6.85	110	12100	9500	14.7
76	5776	4540	7.03	111	12321	9680	15.0
77	5929	4660	7.22	112	12544	9850	15.3
78	6084	4780	7.41	113	12769	10000	15.5
79	6241	4900	7.60	114	12996	10200	15.8
80	6400	5030	7.79	115	13225	10400	16.1
81	6561	5150	7.99	116	13456	10600	16.4
82	6724	5280	8.19	117	13689	10700	16.7
83	6889	5410	8.39	118	13924	10900	17.0
84	7056	5540	8.59	119	14161	11100	17.2
85	7225	5670	8.79	120	14400	11300	17.5
86	7396	5810	9.00	121	14641	11500	17.8
87	7569	5940	9.21	122	14884	11700	18.1
88	7744	6080	9.43	123	15129	11900	18.4
89	7921	6220	9.64	124	15376	12100	18.7
90	8100	6360	9.86	125	15625	12300	19.0
91	8281	6500	10.1	126	15876	12500	19.3
92	8464	6650	10.3	127	16129	12700	19.6
93	<b>8</b> 649	6790	10.5	128	16384	12900	19.9
94	8836	6940	10.8	129	16641	13100	20.3
95	9025	7090	11.0	130	16900	13300	20.6
96	9216	7240	11.2	131	17161	13500	20.9
97	9409	7390	11.4	132	17424	13700	21.2
98	9604	7540	11.7	133	17689	13900	21.5
99	9801	7700	11.9	134	17956	14100	21.8
100	10000	7850	12.2	135	18225	14300	22.2
101	10201	8010	12.4	136	18496	14500	22.5
102	10404	8170	12.7	137	18769	14700	22.8
103	10609	8330	12.9	138	19044	15000	23.2
104	10816	8490	13.2	139	19321	15200	23.5
105	11025	8660	13.4	140	19600	15400	23.8
				A			<b>y</b>

#### APPENDIX VIII

# LIMITATIONS OF THE TECHNIQUES AND SOURCES OF ERROR

#### 1. GENERAL

The results obtained from any test technique can only be of optimum reliability if the technique is followed meticulously by a careful operator. Any deviation from the described procedure can effect the accuracy or precision of the test result. However, the techniques themselves have inherent limitations both in applicability and in the significance of the test results. Some of these limitations are discussed herein, as well as some words of caution about potential sources of error.

# 2. TENSILE TESTS (FMT-1, 2, 3, 4, 14, 15)

# 2.1 Force Measurement

The accuracy of the force measurement depends primarily upon proper calibration of the test instrument. Instructions for this have been provided by the manufacturer, and should be carefully followed. This accuracy also depends, however, upon the proper selection of full-scale load so as to obtain a reasonable pen deflection over the range of loads which are of interest. The load range should always be selected so that the plotted curve extends across at least 1/2 of the chart width.

# 2.2 Extension Measurement

# 2.2.1 Displacement of the measuring system

When extension is measured in terms of an assumed jaw displacement, any displacement of the load measuring system, or slack in the measuring or pulling system, or in the chart drive, will introduce an error in the measurement. Although this is likely to be small, it can be significant when the displacement being measured becomes small.

Example: A 1% displacement for a 1" specimen is 0.01". If the load cell and coupling displacement were as high as 0.005", this would be a 50% error in the assumed displacement.

When extension measurements representing a displacement of the pulling jaw of less than 0.5% are made on a 10 inch specimen, it is essential to realize that the displacement of the measuring system has probably introduced an appreciable error into the measurement, and that sometimes this error can be large relative to the measured extension.

The total displacement of the measuring system can best be determined by inserting in the jaws a linearly elastic specimen having an accurately known modulus. The measured extension at any load can then be compared to a calculated "true" value, and any difference attributed to errors introduced by the measuring system.

# 2.2.2 Specimen Slack

If any slack exists in the specimen when it is mounted in the jaws initially, it will be incorrect to assume that the initial jaw separation is the gage length. This slack can arise because of the presence of crimp, or of a curved specimen, or for any other reason for the straightened length of the specimen being more than the initial jaw spacing.

The presence of slack can be recognized in the shape of the load-elongation curve. If an appreciable amount of jaw movement occurs before the load deviates from zero, this can be used as a measure of the specimen slack, and should be used as a correction to be applied to the gage length (See Appendix I).

For some fibers it may be difficult, if not impossible, to mount the specimen in such a way as to ensure an accurately known gage length. In these cases, it is often desirable intentionally to mount it slack, and to obtain an accurate measure of gage length as outlined in Appendix I.

### 2.2.3 Elevated Temperature

The determination of true gage length in elevated temperature testing is seldom possible, except in the case of the Optical Strain Analyzer. The specimen is mounted at room temperature, using a known jaw spacing. When the temperature is raised, the jaws expand, reducing the distance between them, while the specimen also changes in length (expansion or contraction, depending upon the characteristics of the fibrous material). While these changes are apt to be relatively small, they can be significant when the gage length is short (1 inch). The new "hot" gage length can be determined accurately in the Optical Strain Analyzer by knowing the spacing of the flags at room temperature, and measuring the change in spacing when the temperature is raised, but before load is applied.

# 2.3 Other Influences

All of the testing described in these methods is carried out at fixed gage length and speed, the specific values employed depending upon the instrument being used. It should be recognized, however, that tests done at other gage lengths can give useful information about uniformity of the specimen; and tests at other speeds can provide data which may be more pertinent to specific applications. There can be no best speed of testing. When the material properties are speed-sensitive, as is the case in many of the materials of interest to the Air Force, it is important to study this speed sensitivity in order to predict performance intelligently in any particular end use.

Although provision is made for testing at elevated temperatures, testing at low temperatures can also be important, since many materials are embrittled as the temperature goes down. Many materials are also moisture-sensitive (e.g. glass), so that testing under water at different temperatures, or in steam, can be important. Many such environments can be provided rather easily using existing equipment. Others, such as high vacuum or cryogenic chambers, require rather elaborate equipment and should probably be left to those specialized laboratories which are set up specifically for such work.

### 3. LINEAR DENSITY (FMT-5)

# 3.1 Vibroscope

The Vibroscope provides almost the only way of measuring the linear density of a short length of fine fiber. For most purposes, the errors in this technique can be neglected. However, when an accurate value is required, it may be necessary to make corrections for the stiffness and nonuniformity of the fiber, or for the amplitude of the induced oscillations. These factors are discussed in detail by E. T. L. Voong and D. J. Montgomery (Text. Res. Jour. 23, 821, 1953) and by I. M. Stuart (Brit. Jour. App. Phys. 10, 219, 1959).

3.2 Other techniques are generally more reliable than the Vibroscope when they can be used (i.e. when the fiber is long enough or coarse enough). The greatest source of error is the length measurement, unless only a very small weight of material is used, and this error will be greater for low modulus than for high modulus fibers.

# 4. DIAMETER, CROSS-SECTION (FMT-6)

#### 4.1 Use of the Microscope

Any measurements made with the aid of the microscope must be done very carefully, for significant errors can be introduced by not having the microscope in perfect adjustment. The instructions given in FMT-6 must be followed meticulously when diameter is being measured. The limits of resolution of the microscope for the lenses which are used in this measurement are such that even under the best of circumstances it is unlikely that any result has an accuracy better than about  $\pm 0.01 \text{ mil} (0.25\mu)$ .

# 4.2 Area Determination

If the cross-sectional area is calculated from a diameter measurement, two potential sources of error exist: (a) Any error in the measurement of diameter is approximately doubled, since the diameter must be squared; (b) An assumption of circularity must be made. Both of these errors can be considerable. The direct measure of cross-sectional area from a section is probably more accurate, provided the section image being measured is large enough, but since this cannot be done on the fiber being tested, an assumption of uniformity along the fiber must be made. This can be questionable, particularly for experimental fibers.

### 4.3 Tensile Strength

Any error in the estimate of cross-section results in an error in the calculated tensile strength. Moreover, any nonuniformity along the length of a fiber can affect the breaking strength significantly. This is not adequately compensated for by using an average value for cross-section, even if this is known, to calculate tensile strength.

#### 5. DENSITY (FMT-7)

The accurate measurement of density requires very careful technique. Convection currents, turbulence set up by stirring, nonhomogeneous solutions, air adhering to or trapped by the fiber, absorption of the immersion liquid by the fiber can all influence the result significantly.

#### 6. SHEAR MODULUS (FMT-9)

There are several potential sources of error in a determination of shear modulus by using a torsion pendulum. Some of these are:

6.1 Usually a circular cross-section is assumed, and any departure from circularity will introduce an error in the calculation. When the cross-section is far from circular, as in graphitized viscose, for example, a more reliable approximation of the polar moment of inertia of the fiber should be made.

6.2 A lack of symmetry in the bob due to improper attachment to the fiber will alter its moment of inertia and affect the calculation of modulus.

6.3 Nonuniformity in cross-section along the length of the fiber cannot be compensated for by using an average cross-section. No satisfactory solution to this problem exists, though the modulus is usually calculated by using the average cross-section.

6.4 For some materials, particularly those which are visco-elastic, the value obtained for the shear modulus may be affected by the tension in the fiber (i.e. by the weight of the bob).

6.5 Measurements should always be made using small angular deflections of the bob in order not to exceed the yield shear strain, and since the formula used does not apply for large deflections. If, due to excessive damping of the oscillations, large deflections must be used (corresponding to more than about 1/2 revolution of the bob each side of center) errors may be introduced into the result for short fibers having a low yield strain.

6.6 Bobs should always be made of nonmagnetic materials, since the period of oscillation may otherwise be influenced by the earth's magnetic field, particularly in the case of fibers having a low modulus or a long length, and when the bob is light.

6.7 The torsion pendulum technique cannot be used to measure torque decay at fixed twist, or hysteresis in tors'on, or the effect of continuous cycling, etc. This can be done by other techniques in which the torque developed in the test specimen is balanced against that in another calibrated element such as a standard twisted suspension or a galvanometer movement.

7. BENDING MODULUS -- FLEX JIG (FMT-10)

The primary sources of error in this technique are:

7.1 The accurate measurement of the spacing of the supports.

7.2 Location of the pulling ring exactly midway between the two points of support.

7.3 Suppage of the specimen in the points of support which may occur in stiff specimens, or when the deflection is too large.

7.4 The precise measurement of deflection, particularly as this is affected by the deflection of the measuring system.

8. MODULUS -- VIBRATING REED (FMT-11)

8.1 The accuracy of the technique is largely limited by the sensitivity with which the resonant frequency can be determined. This will depend upon the bending properties of the particular fiber being tested.

9. BENDING MODULUS--SEARLE DOUBLE PENDULUM (FMT-12)

9.1 This method is potentially the most reliable of the three described. Its limitations and sources of error are described fully in the report which is referenced in FMT-12.

10. SONIC MODULUS (FMT-13)

10.1 It is often difficult to determine precisely the position of the successive nodes along the test specimen. Thus, there may be a significant error in the measurement of the wavelength, and in the calculation of the sonic modulus.

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This report describes the technique Nonmetallic Materials Division, Air H the important mechanical and physical density, strength, modulus of elastici procedures described are those in use been in use for some time and a consi has demonstrated the reliability of the be reviewed and updated as required. purpose of: (1) acquainting those conc techniques which have been found to be fibers; and, (2) to provide Air Force used by the Air Force Materials Labo made.	s which are used. Force Materials I I properties of fib ity, and repeated is a the time this is derable amount of e methods. These They are compile erned with fiber p e useful for gather contractors and fiberatory in order th	by the Fibr aboratory, ers and yan flexing prop report was f data have techniques ed and repo property mo ring data on iber users nat valid co	rous Materials Branch, for evaluating some of rns. These include perties. The test prepared. They have been generated which and procedures will orted herein for the easurement with those in a variety of types of with the basic techniques mparisons of data can be	

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The following corrections are applicable to AFML-TR-67-159, "Evaluation Techniques for Fibers and Yarns Used by the Fibrous Materials Branch, Nonmetallic Materials Division, Air Force Materials Laboratory", September 1968.

Page 90

(1) Equation for standard deviation should be changed to read:

$$S = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}}$$

(2) Following equation should be changed to read:

$$S = \sqrt{\frac{n\sum_{x}^{2} - \left(\sum_{x}\right)^{2}}{n(n-1)}}$$

Air Force Materials Laboratory Air Force Systems Command Wright-Patterson AFB, Ohio