REPORT NO. DOT-TSC-RSPA-80-11

FIRE TESTS OF AUTOMOTIVE GRADE CARBON FIBER COMPOSITES

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JUNE 1980

FINAL REPORT

Prepared for

U.S. DEPARTMENT OF TRANSPORTATION
Research and Special Programs Administration
Transportation Programs Bureau
Washington DC 20590

Technical Report Documentation Page

1. Report No. DOT-TSC-RSPA-80-11	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle Fire Tests of Automotive Grade Carbon Fiber Composites		5. Report Date June 1980
		6. Performing Organization Code
7. Author(s) C. Wilton+; J. Boyse+; J. Zaccor+		8. Performing Organization Report No. DOT-TSC-RSPA-80-11
9. Performing Organization Name and Address NASA/Ames Research Center* Moffett Field, CA 94035		10. Work Unit No. (TRAIS) RS015/R0502
		11. Contract or Grant No.
12. Sponsoring Agency Name and Address U.S. Department of Transportation Research & Special Programs Administration Transportation Programs Bureau Washington, DC 20590		13. Type of Report and Period Covered Final Report Mar.1979 - Jan.1979
		14. Sponsoring Agency Code DTS-331
15. Supplementary Notes *In Agreement with U.S. DOT Research and Special Programs Administration Transportation Systems Center Cambridge, MA 02142		+Scientific Services, Inc. 517 East Bayshore Redwood City, CA 94063 Sub-Contractor

16. Abstract

This report presents the results of a fire test study on selected composite materials containing carbon fibers that are planned for use, or that have a high potential for use, in automobiles and other vehicles.

The study objectives were to determine the quantity and distribution of carbon fibers released during exposure of the composites to fires.

The test data indicated that automobile fires, per se, are unlikely to be the cause of serious risk from single-fiber release. However, the possibility of automobile fires occuring in combination with other factors, such as wind and fire fighting, rescue, and disposal operations could result in additional fiber release.

17. Key Words carbon fibers; composites; fire tests; fiber release		18. Distribution Statement DOCUMENT IS AVAILABLE TO THE PUBLIC THROUGH THE NATIONAL TECHNICAL INFORMATION SERVICE, SPRINGFIELD, VIRGINIA 22161	
19. Security Classif. (of this report) unclassified	20. Security Classif. (of this page) unclassified	21. No. of Pages 62	22. Price

Form DOT F 1700.7 (8-72) Reproduction of completed page authorized

Preface

This report presents the results of a fire test study of carbon fiber (CF) composites. The scope of this effort was limited to collecting and counting CF released from burning "automotive grade" CF composites.

The cooperation and valuable guidance of Mr. J. A. Mansfield, NASA/Ames Research Center, is gratefully acknowledged. Appreciation is also expressed to Mr. W. T. Hathaway and Mr. K. M. Hergenrother, Transportation Systems Center for their comments on the final draft of the report. This effort was conducted under the direction of Mr. C. E. Bogner, Technical Monitor, Transportation Systems Center.

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1. INTRODUCTION

This report presents the results of a test program on selected composite materials containing carbon fibers that are planned for use or that have a high potential for use in the future manufacture of automobiles and other vehicles. The primary objective of this program was to determine the quantity of carbon fibers released during exposure of the composites to fires.

This program was conducted for the Department of Transportation by Scientific Services, Inc. under subcontract from NASA Ames Research Center.

TABLE 1. CANDIDATE AUTOMOTIVE COMPOSITES

	Resin Type	Reinforcement Type	Reinforcement Form
TESTED	1. Polyester	graphite/glass hybrid	unidirectional
	2. Polyester	graphite	unidirectional
	3. Polyester	graphite/glass hybrid	cross ply
	4. Vinyl Ester	graphite	unidirectional
	5. Vinyl Ester	graphite/glass hybrid	cross ply
	6. Epoxy	graphite	unidirectional
NOT TESTED	7. Polyester	graphite/glass	chopped
	8. Epoxy	graphite	cross ply
	9. Polyester	graphite	woven

by the flame and blocked by a comparable radiation from the flame, with the result that the final temperature of the structure approaches the temperature of the flame. This flame exposure condition is designated the "High Radiant" condition.

It was anticipated that these two exposure conditions should bracket the range of fire exposures expected to occur in automotive type fires. Oxygen-to-fuel ratios could also be expected to vary; therefore, a range of ratios was included in the test program.

Flame and hot combustion gases may impinge on the face or on the edge of a component, or both the face and edge simultaneously. This investigation was restricted to edge impingement, as this was assumed to be the condition that would result in the greatest fiber release; i.e., the worst case condition.

The final factor receiving attention as part of this study to achieve a realistic assessment of potential graphite fiber release was the post-fire environment. An automobile fire, having been extinguished or having burned out, is likely to leave weakened fiber residue exposed to many adverse environmental conditions that will cause further deterioration. These conditions include winds that may break loose fibers from a burned component, mechanical stress due to structural damage to the automobile body, and mechanical and vibrational stress occurring when a burned automobile is removed from the accident scene. A limited investigation was conducted in this area which consisted of exposure of a burned and cooled sample to 6 and 12 mph winds, physically damaging the sample (flexural buckling) and re-exposing it to a 12 mph wind.

A summary of the tests performed is presented in Table 2.

TEST	SAMPLE DESCRIPTION	BURN CONDITIONS						NOTES
		Low Rad	High Rad	Fuel Rich	O_2 Rich	Burn Time	Vel ft/s	
14	Vinyl Ester - Graphite (AS-5)/glass hybrid cross ply		X		X	10	10	
15	Vinyl Ester - Graphite (AS-5)/glass hybrid unidirectional		X		X			
16	Polyester - Graphite (AS-5)/glass hybrid unidirectional	X		X				
17	Polyester - Graphite (AS-5)/glass hybrid unidirectional	X		X				
18	Polyester - Graphite (AS-5)/glass hybrid unidirectional	X		X				
19	Polyester - Graphite (AS-5)/glass hybrid unidirectional		X	X				
20	Repeat of Sample 18	na	na	na	na	na		(4)
21	Repeat of Sample 19	na	na	na	na	na		(5)
22	Repeat of Sample 19	na	na	na	na	na		(6)
23	Repeat of Sample 19	na	na	na	na	na		(7)

Notes:

(1) Aircraft grade sample — used for system checkout only.

(2) Experimental grade sample — used for system checkout only

(3) Burned with outside fiber layer horizontal instead of vertical

(4) Sample 18 at 6 mph

(5) Sample 19 at 6 mph

(6) Sample 10 at 12 mph

(7) Sample 19 — broken — at 12 mph

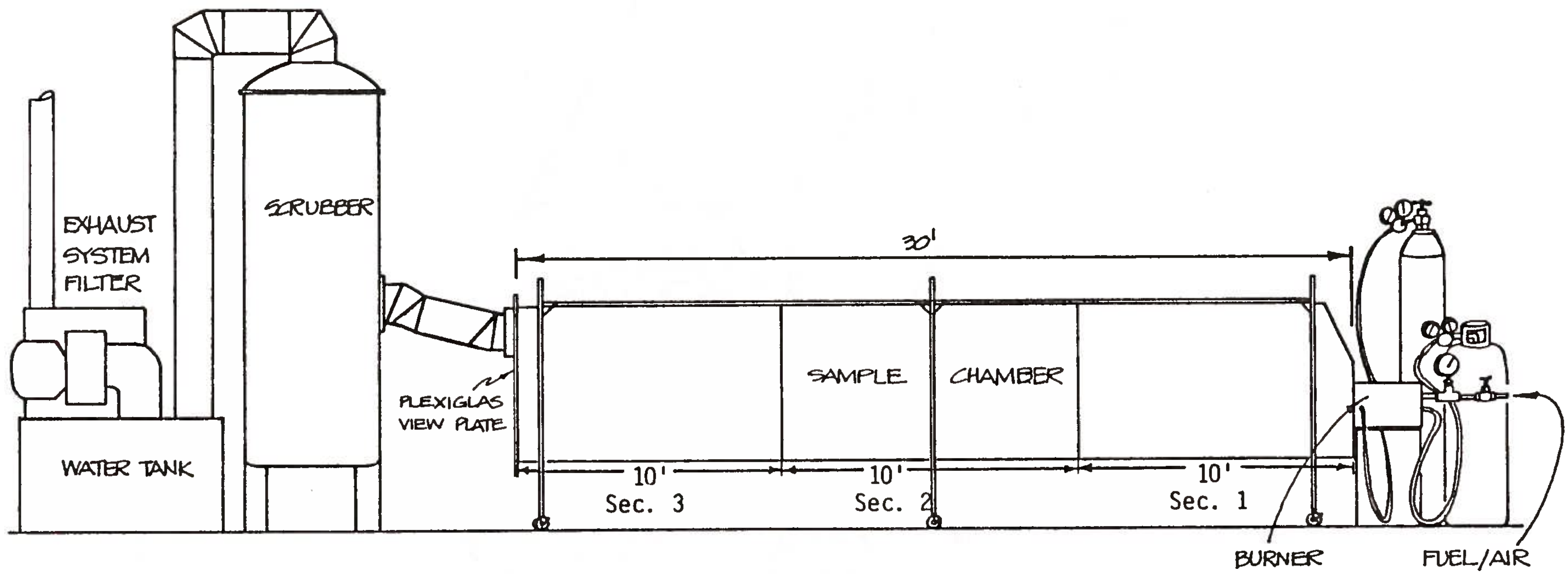
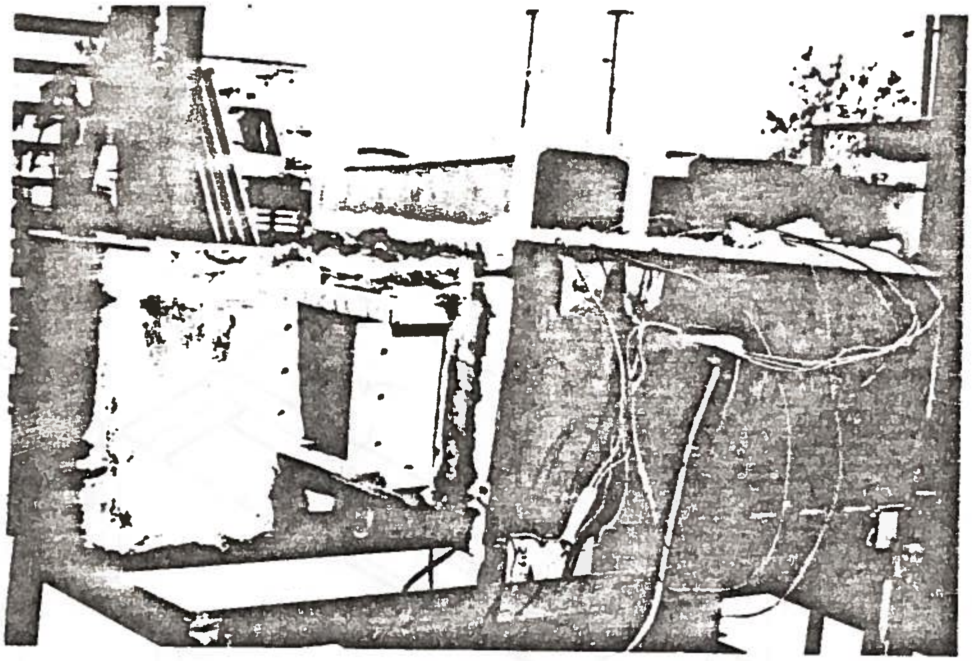


Fig. 1. Sketch of Test Facility.



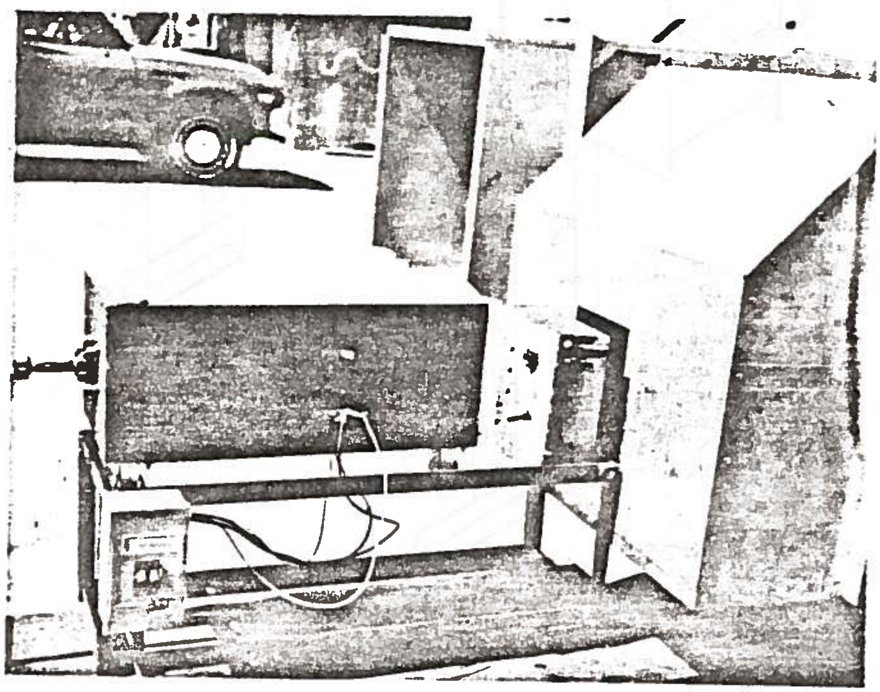


Fig. 3. Photographs of High Radiant Burn Chamber.

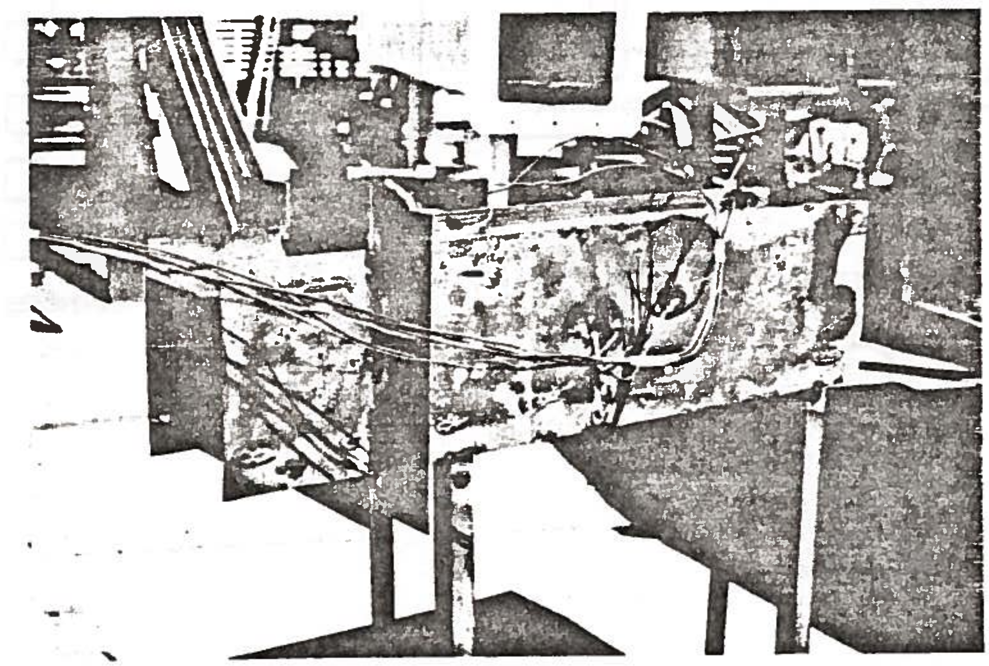


Fig. 5. Photograph of Low Radiant Burn Chamber.

Both burn chambers were designed to be inserted into the sample collection chamber (described below) to a depth of three inches and sealed. The high-radiant input burn chamber was sealed against the opening in the collection chamber by the insulation material. For the low-radiant input burn chamber, a collar was constructed three inches from the end for this purpose (see Figs. 4 and 5).

The burn chambers and collection duct were equipped with thermocouples to monitor flame temperature and preheat temperature and to assure that proper cooling was taking place in the collection duct. The positions of the thermocouples in the burn chambers are shown in Fig. 7.

3.2 SAMPLE COLLECTION CHAMBER

The purpose of the sample collection chamber was to collect all graphite fibers released from the burned samples. The theory was to create a chamber big enough in cross-section to reduce the flow velocity from the burn chamber and long enough to allow all the graphite particles to settle out before reaching the far end.

The design criteria required a maximum flow velocity of 15 ft/sec through the burn chamber. A maximum flow velocity of 15 ft/sec within the burn chamber would be reduced within the collection chamber — which had a cross-section of 33 in. x 33 in. (1,089 sq in.) — by a factor of almost 44, and would be further reduced by cooling along the length of the duct. The aerodynamic model that was applied required that a duct with this cross-section be 30 ft long to provide adequate time for all fibers to fall to the floor before reaching the end. Once on the floor, the fibers were collected and counted by a method described in the "Fiber Collection and Counting Procedure" section of this report.

The sample collection chamber was constructed of 16-gauge stainless steel sheet and was made in three 10-ft sections. Each duct was supported by a steel framework and mounted on casters. The side seams were riveted together and sealed with tape. During a test run, the sections were clamped together with vice grips and sealed with tape.

At the burn chamber end of the duct was a short section with one end having an 8 in. x 8 in. hole into which the burn chamber was inserted, and with the other end expanding to the full duct dimension of 33 in. x 33 in. At the opposite end of the duct, the exhaust end was closed off by a 3/8-in. thick Plexiglas sheet, which provided a view down the duct to the burn chamber.

3.3 SCRUBBER FILTER SYSTEM

The hot gases exiting from the collection duct were passed through a filter system to remove fibers. This system included a Hepa filter, a water scrubbing system, and a post-scrubber filter system. The scrubber filter system is further described in an SSI report entitled "Fire Testing NASA Samples — Phase I", February 1979.

The first filter of the system was affixed directly to the Plexiglas at the end of the fiber collecting duct (see Fig. 1). Rigid frames held filter paper against a wire screen support. The filter frame was inserted into an open-ended box which was bolted to the end of the duct. The filter frames were sealed against the front and back of the box with felt. With this method, filters could be changed during the experiment to prevent a severe reduction of the exhaust rate caused by clogging of the filters with unburned residue from the sample. The first filter system also provided a means to determine if any fibers failed to settle to the floor.

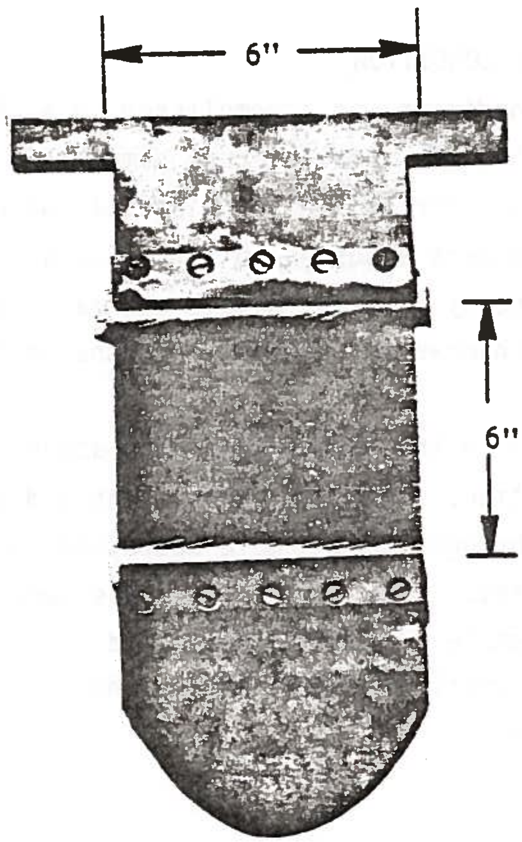


Fig. 8. Photograph of Sample Holder.

5. FIBER COLLECTION AND COUNTING PROCEDURE

After the duct had been cleared of smoke and allowed to cool, the clamps holding the sections together were removed and the sealing tapes slit at each junction so that thin aluminum sheets could be inserted before the sections were separated. The aluminum sheets sealed each duct section from local air currents that might disturb or expel fibers. These sheets, which had small sliding ports in them through which the fibers were collected, were clamped to the duct section ends with vice grips and taped around the edges to seal them. With each of the three sections completely sealed, the center section was then removed.

Fibers were collected from each section by means of a 4 in. x 15 in. adhesive-coated, translucent paper affixed to a cylinder roller which was attached to a long handle. This roller was inserted through the sampling ports and the sticky surface rolled over the fibers to collect them. For a given test, the area that could be rolled with a single piece of adhesive paper was limited by the quantity of soot deposited along with the fibers. The density of soot on the floor was usually greater than that of the fibers and significantly reduced the effectiveness of the adhesive after collecting from the bottom surface of each duct section.

The area that could be rolled with a single strip of adhesive paper to collect a suitable sample for analysis was also limited by the density of the fibers. This limit was imposed by the fiber-counting technique rather than the efficiency of the adhesive. Early in the experimental program, a single piece of adhesive paper was used to collect from only a few square feet of the duct floor. The resultant low density of fibers on the adhesive paper required that a large area of adhesive paper be examined carefully before a statistically significant number of fibers

In most cases, the adhesive paper sheets were not examined in their entirety, but subdivided into strips that ran completely across the adhesive (perpendicular to the direction in which it was rolled) and then randomly selected for counting. Subdividing across the adhesive strip ensured no bias in the count due to the rolling process used in collecting the fibers. To be complete, the fiber collection process required overlapping of previously rolled areas so that all fibers would be exposed to the adhesive. Thus, at the edges of the tape, the density of fibers was expected to be lower than in the middle of the tape because of overlapping a region already collected.

In all cases, the entire collecting surface was rolled with adhesive. Follow-up tests of the collection efficiency were conducted by repeating the collection process and comparing counts. These showed less than ten percent of the fibers were missed in the first pass.

Assessments were also made to determine how many fibers passed through the 30 ft of duct without falling to the floor. This simply required microscopic examination of the paper filter at the downstream end of the duct. Very few fibers were ever observed on this filter. Moreover, the walls and ceiling of the duct were sampled with the same adhesive roller system and these samples were examined under the microscope. In this case, few fibers were observed. Thus, the testing and collecting procedures were found to provide samples with counts not more than ten percent low.

Recounts of the same area of a test sample by the same and by different observers showed variations within ±33%. Counts of different areas of the same test sample showed variations in assessment of the total count to be similar in magnitude. Thus, variations of ±33% between test samples cannot be considered significant. The size-frequency distributions in different sections of the duct were examined and found to be essentially the same (though counts in these sections varied considerably sometimes). These assessments showed the procedures used in these tests were more than adequate to discern differences of 100% or more by means of a single test.

TABLE 3A. NUMBER OF FIBERS RELEASED

Fiber Orientation				Unidirectional				Cross Ply	
Glass Content				YES		NO		YES	
Fiber Type				AS 5	GLC	AS 5	GLC	AS 5	GLC
Oxygen Rich	High Radiant Input	Binder	Vinyl Ester	* 15 - 4,125				11 - 11,535 12 - 19,602 14 - 43,182 3 - 947 †	
Oxygen Rich	High Radiant Input	Binder	Epoxy			6 - 41,486 8 - 22,806			
Oxygen Rich	High Radiant Input	Binder	Poly-Ester	13 - 3,390	4 - 1,883	5 - 56,983	7 - 9,296	9 - 13,046 10 - 11,415	
Fuel Rich	Low Radiant Input	Binder	Polyester	16 - 319,260 17 - 126,339 18 - 44,239					
Fuel Rich	High Radiant Input	Binder	Polyester	19 - 1,351					

* Test number in ○

† Sample was oriented with the outside layer of fibers horizontal; in all other cases this layer was vertical.

TABLE 4. TEST NUMBERS AND CORRESPONDING FIGURE NUMBERS

Figure Number	Test Number
9	15
10	11, 12, 14
11	3
12	6, 8
13	13
14	4
15	5
16	7
17	9, 10
18	16, 17, 18
19	19

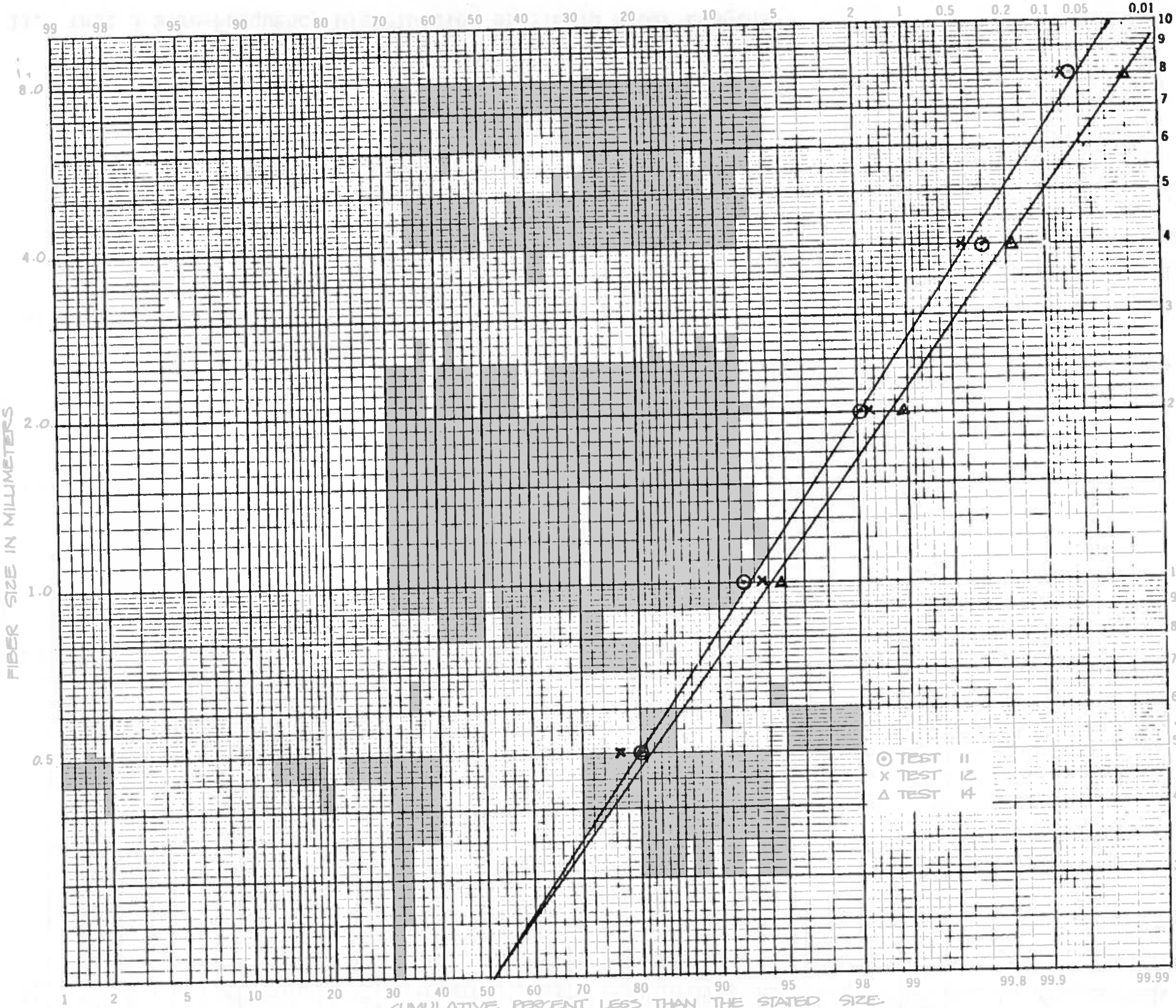


Fig. 10. Test 11, 12, and 14 Size-Frequency Distribution of Single Fiber Lengths.

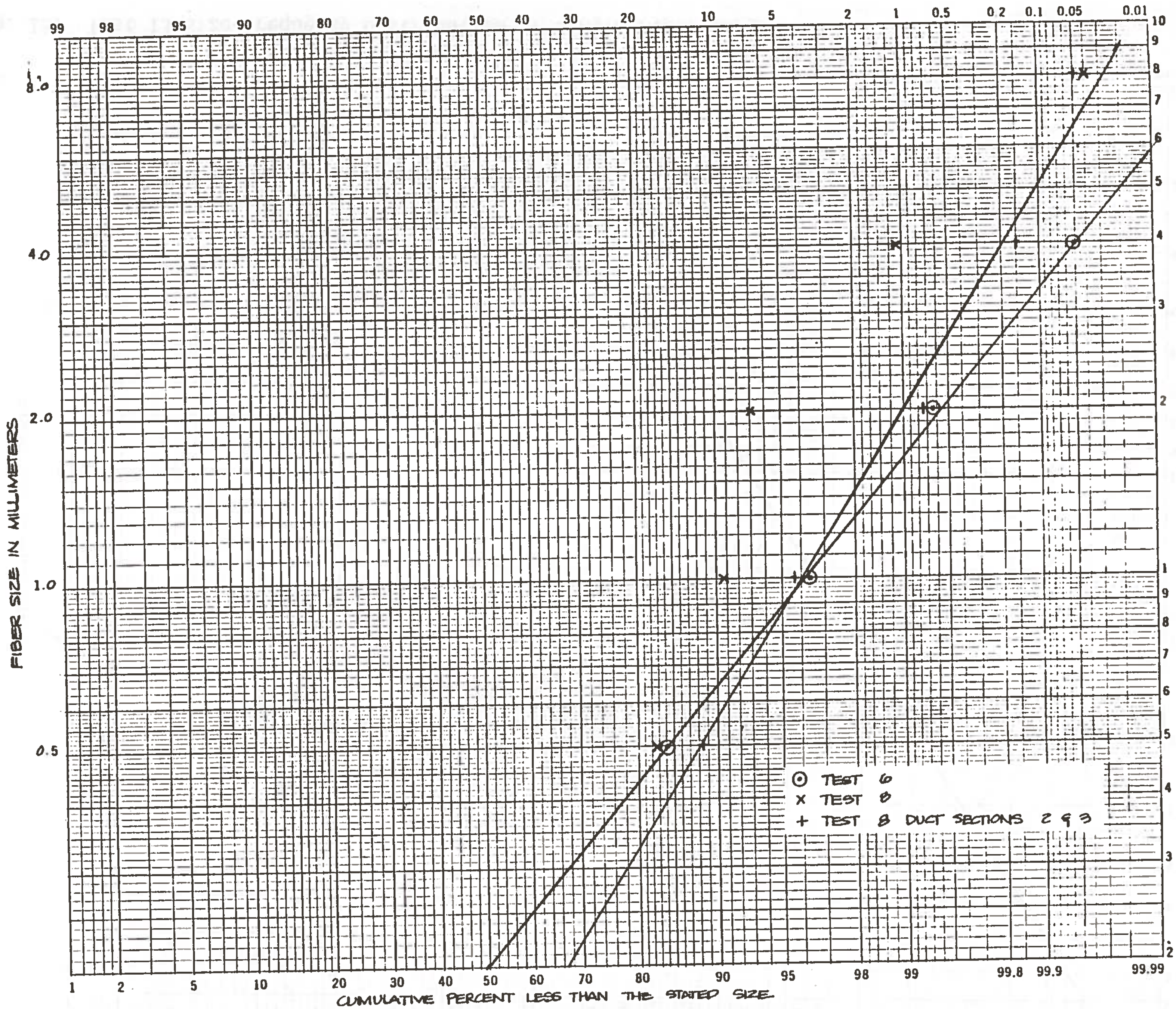


Fig. 12. Test 6 and 8 Size-Frequency Distribution of Single Fiber Lengths.

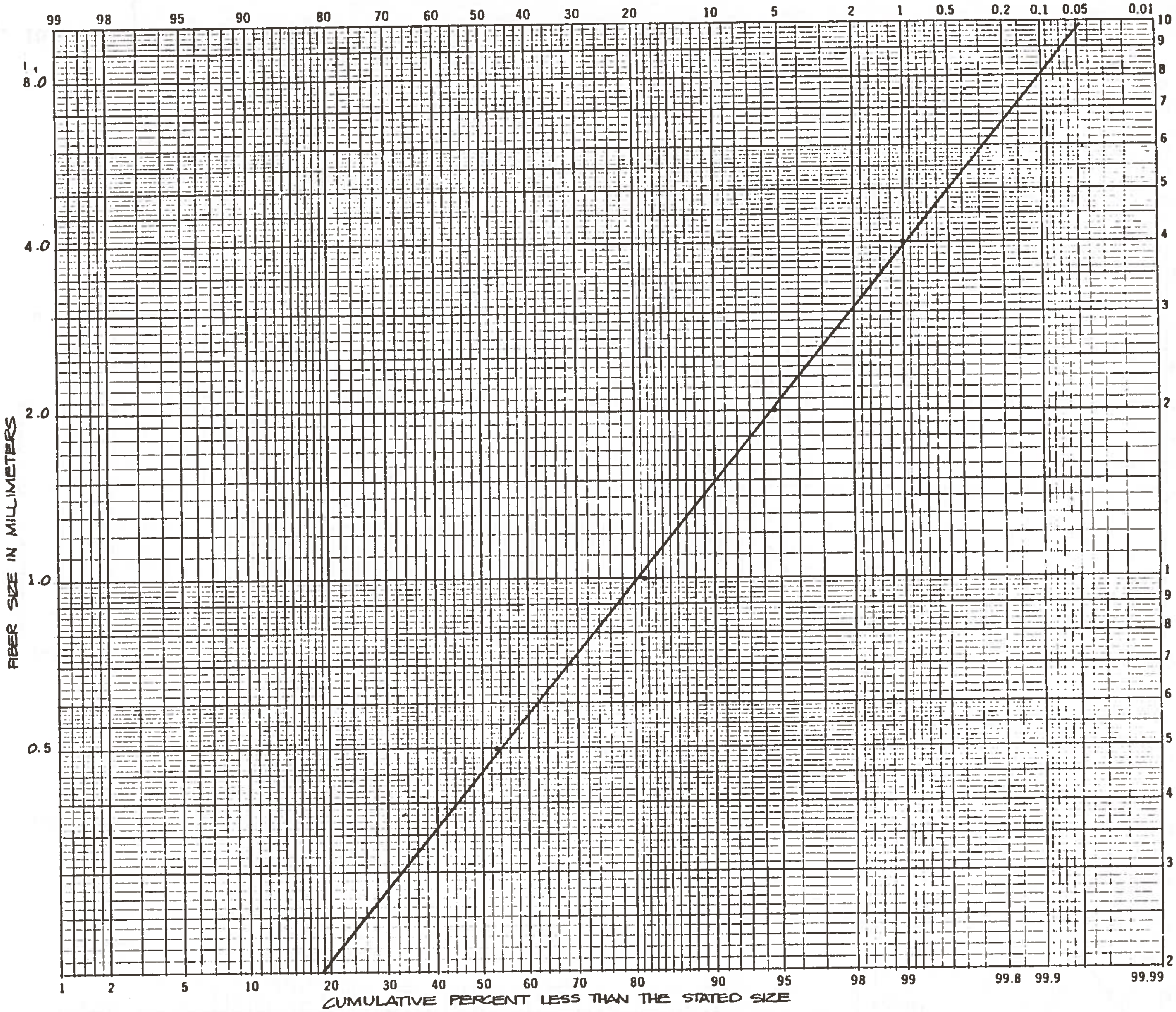


Fig. 14. Test 4 Size-Frequency Distribution of Single Fiber Lengths.

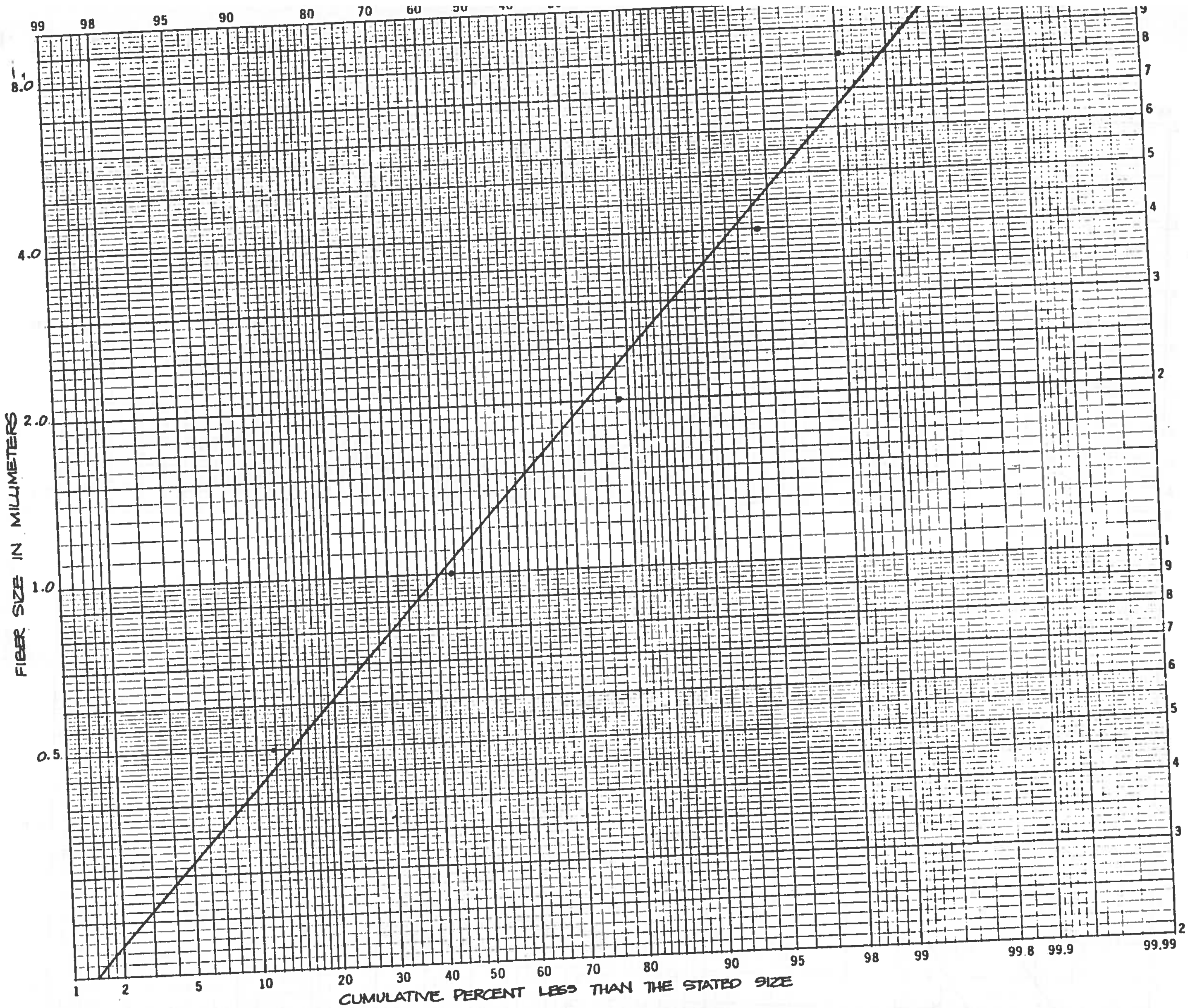


Fig. 16. Test 7 Size-Frequency Distribution of Single Fiber Lengths.

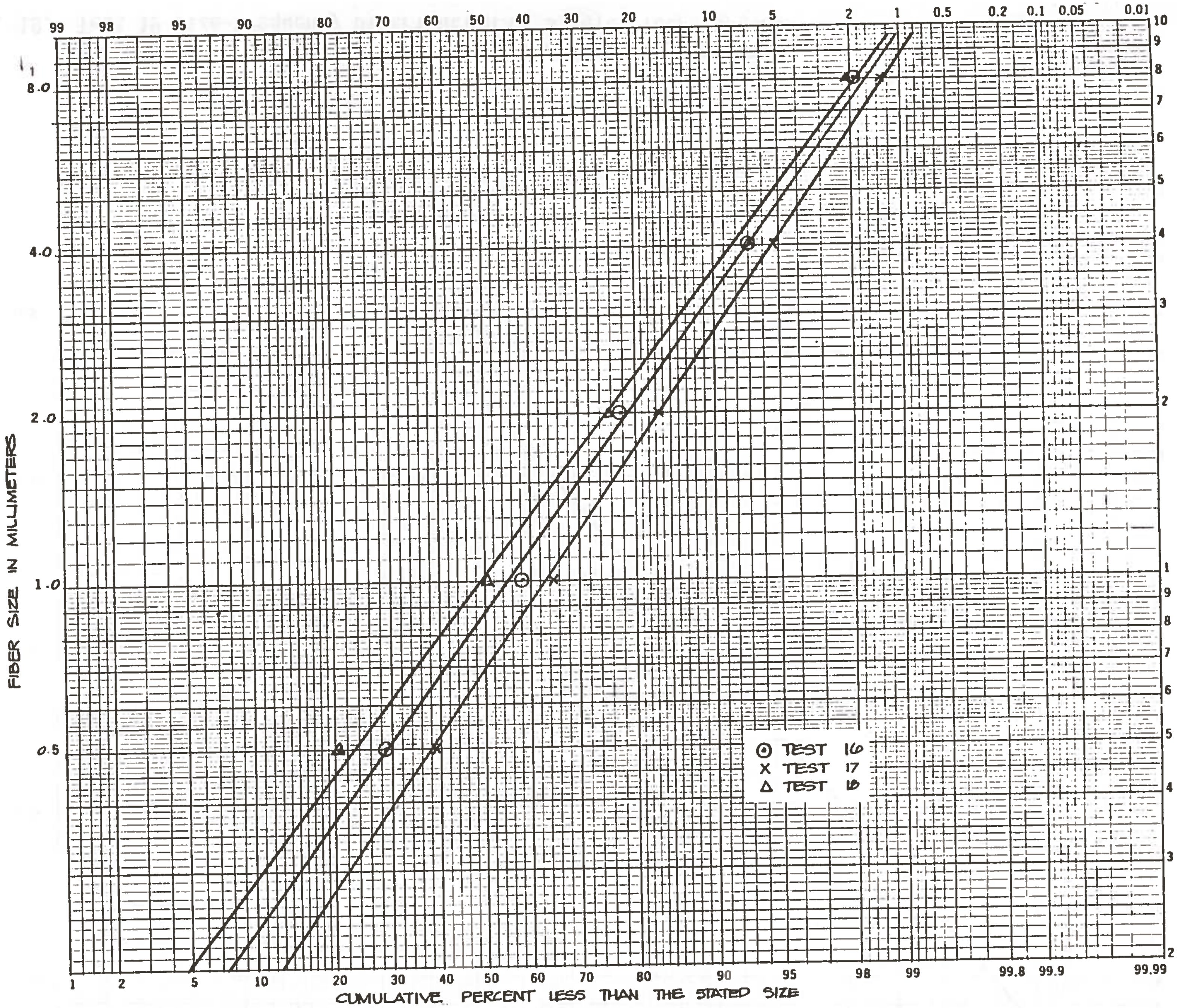


Fig. 18. Test 16, 17, and 18 Size-Frequency Distribution of Single Fiber Lengths.

cause; when the section 2 and 3 data are taken alone, they are reasonably well behaved. It is believed that a tuft of fibers may have been formed in this test and released to bias the data, near the burn chamber, towards larger fiber sizes.

Regarding the present study of potential hazards of automotive grade graphite composites, where the observed differences may be considered significant depends entirely on the purpose or objective of the analysis. At this stage, large differences on the order of 300% to 600% would be of great interest; differences on the order of 100% to 300% would be of considerable interest; differences of 30% to 100% would be of some interest; and differences of less than 30% would be of little interest (because the latter would be submerged in the experimental variation in counting and sizing procedures). Thus, the data in all the multiple plots may be considered, for our present purposes, identical.

It is of interest that the corresponding single-fiber release data for Tests 11, 12, 14, and for Tests 16, 17, 18 also follow log normal probability distributions. These are plotted in Figs. 20 and 21. Both the size-frequency distributions and the single-fiber release data from Tests 11, 12, and 14 obviously belong to a single set, and those from Tests 16, 17, and 18 belong to another. Thus, real differences exist between tests of different types and are detectable in the data for size-frequency distribution and quantity of fibers released.

The two sets of data in Figs. 20 and 21 are limited (only three tests each), but they can be applied to demonstrate how a statistically significant number of tests can be used to make estimates of probability of single-fiber release in excess of some "acceptable" number, for the conditions represented in the testing. As an example, the data in Fig. 21 have been extrapolated to the 99.9 percentile rank to compute that there is one chance in a thousand the count would ever exceed twelve million single fibers released under conditions that correspond to parameters used in these tests.

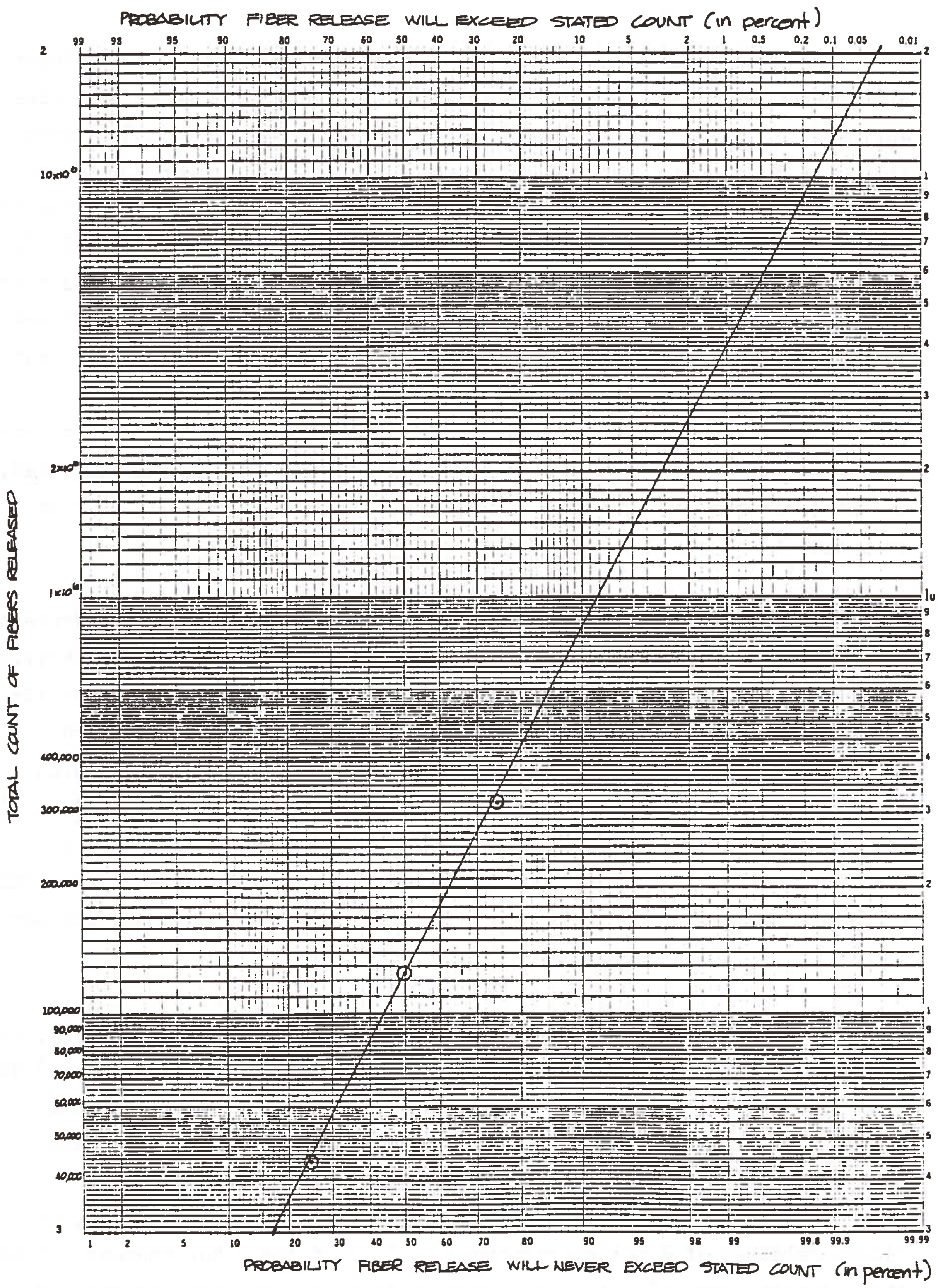


Fig. 21. Fiber Release Data Typical of AS-5 - Unidirectional - Glass Hybrid-Polyester.

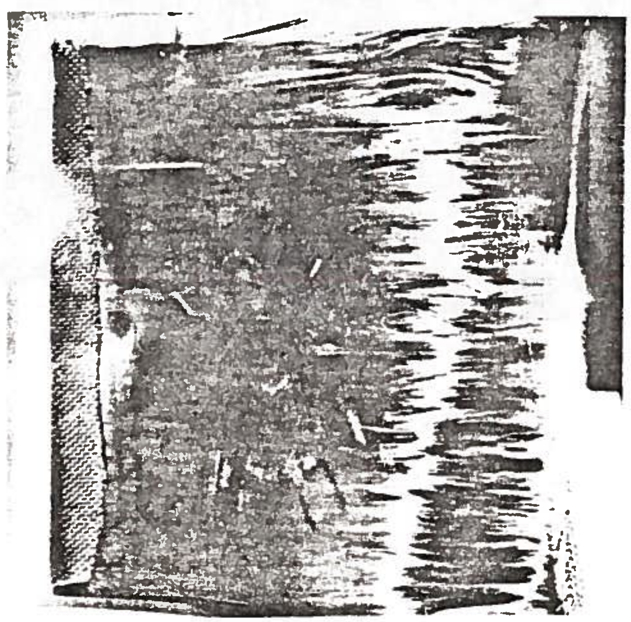


Fig. 22. Sample with Plastic Binder Removed.

again, but increased to about 12 mph and continued for 10 minutes. Additional fuzz balls of fiber were collected and weighed. This time the weight of fibers corresponded to an additional 1.3%. It may be assumed that at an initial wind loading of 12 mph, the combined total would have been released.

If it is typical for winds to exist that could disrupt fibers, it is certain that the process of fighting the fire and collecting the debris will introduce even greater stresses that could cause fibers to be released. If the composite were a support bracket, the weight alone could crack the damaged material in two. To simulate this condition, the above sample was damaged by dragging a hooked rod through the middle, severing all the fibers, and then the gas flow rate of 12 mph was applied for 10 minutes to simulate a wind stream blowing on the damaged fibers. The result was an additional release of 9.5%. Thus the aggregate release for this credible condition corresponded to 3.1% + 1.3% + 9.5%, or 13.9%.

To determine if the burned fibers were still conductive, a number of resistance measurements were taken at random on both the burned samples and on some of the collected fibers. All were found to be still conductive.

The overall conclusions from these tests are that automotive fires, per se, are unlikely to be the cause of serious risk from single-fiber release. However, the possibility of automobile fires occurring in combination with other factors, such as wind and fire fighting, rescue, and disposal operations could result in additional CF release.

APPENDIX A

TEST SAMPLE COMPOSITION

HITCO

GARDENA, CALIFORNIA

CUSTOMER NASA/Ames
ORDER NO. A67290B EAF
PART NO. Crossplied UMC-8057/790
PART NAME Vinyl Ester/Graphite
HITCO S/O NO. Glass Hybrid

Test Number(s) 3, 11, 12, 14

Vinyl Ester/Graphite/Glass Hybrid - Crossplied

Dow Chemical - Derekane 790	25%/wt
Hercules-AS-5 Graphite - Crossplied	30%/wt
Owens Corning or Pittsburgh Corning E Glass	45%/wt

Cure

(a) 300° F. - 1 hr - 300 psi

(b) No Post Cure

Layup Sequence

Graphite/Glass/Graphite/Glass/Glass/Graphite/Glass/Graphite

HITCO

GARDENA, CALIFORNIA

CUSTOMER NASA/Ames
ORDER NO. A67290B EAF
PART NO. Unidirect. AS-5/14029
PART NAME Polyester/Graphite
HITCO S/O NO. 144450 Item 010

Test Number(s) 5

Polyester - Graphite - Unidirectional

U.S.S. Chemical Co. #14029	40%
Hercules AS-5 Graphite	60%

Cure

(a) 300° F. - 1 hr - 300 psi

(b) No post cure

HITCO
GARDENA, CALIFORNIA

CUSTOMER NASA/Ames
ORDER NO. W.O. 9353
PART NO. Experimental Compound
PART NAME Polyester/Graphite
HITCO S/O NO. Unidirectional

Test Number(s) 7

Polyester - Graphite - Unidirectional

Great Lakes Carbon Graphite H-40
& Hercules Thornall # 300 60%/wt

U.S.S. Chemical Company #14029 40%/wt

Cure

(a) $375^{\circ}F$ - 2 hr - 200 psi
$300^{\circ}F$ - 1 hr - 300 psi

(b) No Post Cure

Layup Sequence

3 Thornal/2 H-40/4 Thornal/2 H-40/3 Thornal

HITCO
GARDENA, CALIFORNIA

CUSTOMER NASA/Ames
ORDER NO. Work Order 9353
PART NO. Unidirectional UMC-8057
PART NAME Polyester/Graphite/
HITCO S/O NO. Glass Hybrid

Test Number(s) 13, 16, 17, 18, 19

Polyester - Graphite/Glass Hybrid - Unidirectional

U.S.S. Chemical Co. #14029	25%
Hercules AS-5 Graphite - Unidirectional	30%
Owens Corning or Pittsburgh Corning E Glass	45%

Cure

(a) 300° F. - 1 hr - 300 psi

(b) No post cure

Layup Sequence

Graphite/Glass/Graphite/Glass/Glass/Graphite/Glass/Graphite

APPENDIX B

REPORT OF NEW TECHNOLOGY

After a review of the work performed under this contract, no new innovations, discoveries, or inventions were made or patents submitted. The project did result in a better understanding of the amounts and characteristics of the carbon fiber released from automotive grade carbon fiber composites exposed to automobile fires.