NASA CR- 159,217

NASA-CR-159217
19800012969

NASA Contractor Report 159217

EVALUATION OF MICRON SIZE CARBON FIBERS RELEASED FROM BURNING GRAPHITE COMPOSITES

B. SUSSHOLZ

TRW Defense and Space Systems Group
Redondo Beach, CA 90278

CONTRACT NAS 1–15465

APRIL 1980

NASA
National Aeronautics and
Space Administration

Langley Research Center
Hampton, Virginia 23665
AC 804 827-3966

NASA Contractor Report 159217

EVALUATION OF MICRON SIZE CARBON FIBERS RELEASED FROM BURNING GRAPHITE COMPOSITES

B. SUSSHOLZ

TRW Defense and Space Systems Group
Redondo Beach, CA 90278

CONTRACT NAS 1–15465

APRIL 1980

National Aeronautics and
Space Administration

Langley Research Center
Hampton, Virginia 23665
AC 804 827-3966

N80-21454 #

CONTENTS

TABLES

FIGURES

FIGURES

EVALAUTION OF MICRON SIZE CARBON FIBERS RELEASED FROM BURNING GRAPHITE COMPOSITES

B. SUSSHOLZ

TRW Defense and Space Systems Group

SUMMARY

Quantitative estimates are presented of micron carbon fibers released during the burning of graphite composites. The analyses were based principally on experimental data obtained during large pool fire tests with spoiler and cockpit composite structures conducted at the Naval Weapon Center at China Lake. Fibrillated particles constituted the predominant source of the micron fiber data. Evidence of fibrillation effects due to fiber oxidation by the fire environment was obtained by means of scanning electron microscopy.

Data reduction and analyses were performed of representative records from NASA propane burn tests of composite samples. Results indicated that considerable fiber oxidation occurred in cases where a wind source was activated immediately following termination of the propane burn whereas relatively insignificant oxidation occurred during normal propane burn tests.

It was estimated that the total number of micron fibers generated per kilogram mass of carbon fiber released during an aircraft accident would be about 5×10^{11}, with a mass fraction of 5 percent of the total fiber released. An extreme case analysis was performed to establish upper bounds to the micron carbon fiber concentration level and exposure level downwind from an aircraft accident. Results indicated the upper limit of the micron carbon fiber concentration level was only about half the permissible OSHA asbestos ceiling concentration level.

1. INTRODUCTION

Quantitative estimates are presented herein of the dimensions and frequency of occurrence of small-diameter carbon fibers released from burning graphite composites as a frame of reference for comparisons with criteria established for known fiber hazards. Development of the data was based principally on data reduction and analysis of sticky paper records from pool fire tests of carbon composite structural elements at the Naval Weapons Center (NWC) at China Lake. Additional information was gained by evaluation of carbon fibers deposited within Petri dish samplers during similar tests at the U.S. Army Dugway Proving Ground (DPG). An evaluation was performed by means of analysis of representative NASA sticky paper records from tests at the Naval Surface Weapons Center (NSWC), Dahlgren, VA., of the occurrence of micron size carbon particles for the case of propane burner chamber tests of spoiler and plate carbon composite samples.

Use of commercial products or names of manufacturers in this report does not constitute official endorsement of such products or manufacturers, either expressed or implied, by the National Aeronautics and Space Administration.

2. SYMBOLS

C	fiber concentration level, f/m^3
D	fiber diameter, μm
$\overline{D}$	average fiber diameter, μm
D_S	fiber plume source diameter, m
E	fiber exposure level, $f\text{-}s/m^3$
L	fiber length
$\overline{L}$	average fiber length
$\overline{M}$	average fiber mass, g
M_T	total fiber mass, g
N_f	fiber number
t_b	burn time, s
V_D	fiber deposition velocity, m/s
V_W	wind velocity, m/s
ρ	fiber mass density, g/cc
ρ_{PD}	areal density for Petri dish record, f/m^2
ρ_{BV}	areal density for bridal veil record, f/m^2
ρ_{SP}	areal density for sticky paper record, f/m^2

ABBREVIATIONS

DPG	Dugway Proving Ground
NSWC	Naval Surface Weapons Center
NWC	Naval Weapon Center
OSHA	Occupational Health and Safety Administration
PAN	polyacrylonitrile
T	Thornel
TC	thermocouple
TGA	thermal gravimetric analysis

3. STUDY GUIDELINES

Principal objectives of the study may be briefly summarized as follows:

- Determine the relative amounts of single fibers generated during burn tests of graphite composites of such dimensions as to be potentially respirable by humans.

- Quantify the extent of occurrence of small-diameter carbon particles as a frame of reference for comparisons with criteria established for known fiber hazards.

During the initial phase of the study attention was to be focussed on an evaluation of the passive instrumentation records from NWC Test 11[1] to determine the nature of the existence of carbon fibers in the range of 1 to 2 microns in diameter and 3 to 20 microns in length. In the event evidence was established of the occurrence of carbon fibers in this micron size domain then consideration was to be directed toward characterization of the particle distributions in terms of diameter, length and quantities.

During the data reduction of the sticky paper records the presence was noted of composite groups of fibers which propagated as a collective integrated unit and which were individually of relatively small diameters. A question arose as to whether these particle clusters originated by oxidation of multiple adjacent fibers with corresponding diameter reductions or possibly were caused by fibrillation of a single parent fiber.

An investigation of this question was to be performed during the second phase of the study. The evaluation of pertinent NWC test data in this regard was to be supplemented by a study of representative samples of carbon fibers collected in shallow Petri dishes during pool fire tests of graphite composite samples at the Dugway Proving Ground[2].

In addition to the above considerations, data reduction and analyses were to be performed of representative sticky paper records of NASA tests AF-4, AF-6 and BT-244 conducted at NSWC/Dahlgren Laboratory for the purpose of determination of the single fiber length vs single fiber diameter spectrum for all lengths of single fibers, with the primary objective of quantifying the relative amounts of single fiber generated which are of such dimensions as to be categorized in the small-diameter domain of particular interest.

4. BACKGROUND CONSIDERATIONS

An effort was made to establish appropriate diameter and length bounds for carbon fibers that may be potentially respirable by humans and of interest toward establishing fiber hazard criteria. There is considerable evidence that durable fibers of similar ranges of dimensions constitute health hazards simply because of physical properties rather than chemical nature. Extensive studies have been performed regarding health hazards associated with fibers such as asbestos and glass with very limited research relative to carbon fibers. In consideration of potential carbon fiber hazards it has been generally recommended that due to a lack of other specifically developed guidelines the existing framework of information on asbestos and glass fibers may be applied toward establishing bounds on carbon fiber criteria.

An overview of the current state of the art indicated a range of exposure levels requiring consideration in the evaluation of carbon fiber hazard criteria. A comparison of exposure criteria from various sources(4-11) is presented in Table 1. Over recent years the Occupational Safety and Health Administration (OSHA)(4,5) permissible concentration level for asbestos had been reduced from 5 fibers/cc to 2 fibers/cc for an 8-hour time weighted average, with a proposed(6) further reduction to 0.5 fibers/cc. The corresponding value recommended by the National Institute for Occupational Safety and Health (NIOSH)(7) is 0.1 fibers/cc based on a rationale for establishing a standard that is set at the lowest level detectable by available techniques, with the recognition that evaluation of all available human data provides no evidence for a threshold or "safe" level of exposure.

It is of interest to note that the NIOSH recommended exposure criteria for fibrous glass(8) is 3 fibers/cc, or a factor of 30 greater than for asbestos, indicating relative sensitivities to different types of fiber. Other examples of recognized differentiation are evident from the Threshold Limit Values proposed by the American Conference of Governmental Industrial Hygienists (ACGIH)(9,10) for several types of asbestos ranging from 2 fibers/cc to 0.2 fibers/cc, as well as the results of the study by the British Advisory Committee for the Health and Safety Commission(11) yielding a spectrum of recommended values of 1 fiber/cc to 0.2 fibers/cc for the same set of asbestos types

Size and shape of the fibers are important factors, with significant data indicating that fibers less than 0.5 microns in diameter are most active toward producing certain kinds of damage. Exposure criteria are generally designated for fibers with diameter less than 5 microns, length greater than 5 microns and length to diameter ratio of at least 3. Upper limit estimates of length are of the order of 50 to 100 microns.

Asbestos and glass fibers are of relatively uniform diameter. For the case of carbon fibers released by burning graphite composites there occur many fibers with significant variations in diameter due to differiential oxidation effects over the length of the fiber, with minimum diameters generally at the fiber ends. A question arose as to the appropriate diameter values in relation to

Table 1. Exposure Criteria Comparison

SOURCE	DATE	MATERIAL	CONCENTRATION LEVEL[1] (FIBERS/CC)	FIBER LENGTH (MICRON)	FIBER DIAMETER (MICRON)	LENGTH TO DIAMETER RATIO
OSHA						
STANDARD	JULY 1972	ASBESTOS	5	> 5	–	⩾ 3
STANDARD	JULY 1976	ASBESTOS	2	> 5	–	⩾ 3
PROPOSED	OCT 1975	ASBESTOS	0.5	> 5	⩽ 5	⩾ 3
NIOSH						
RECOMMENDED	DEC 1976	ASBESTOS	0.1	> 5	–	⩾ 3
RECOMMENDED	APR 1977	FIBROUS GLASS	3	>10	⩽ 3	⩾ 3
ACGIH[2]						
TLV[3]	1978	ASBESTOS	5	> 5	–	⩾ 3
PROPOSED	1978	ASBESTOS				
		CHRYSOTILE	2	> 5	–	⩾ 3
		AMOSITE	0.5	> 5	–	⩾ 3
		CROCIDOLITE	0.2	> 5	–	⩾ 3
BACA[4]	OCT 1979	ASBESTOS				
		CHRYSOTILE	1	10 – 80	0.5 – 2.5	⩾ 3
		AMOSITE	0.5	10 – 80	0.5 – 2.5	⩾ 3
		CROCIDOLITE	0.2	10 – 80	0.5 – 2.5	⩾ 3

(1) 8-HOUR TIME WEIGHTED AVERAGE

(2) AMERICAN CONFERENCE OF GOVERNMENTAL HYGIENISTS

(3) TOLERANCE LIMIT VALUES

(4) BRITISH ADVISORY COMMITTEE ON ASBESTOS FOR HEALTH AND SAFETY COMMISSION

development of exposure criteria. For the purpose of the present study average diameter values were assumed.

Based on the foregoing considerations the following baseline values were assumed for the data reduction and analyses:

- Dimension Limits
  - Diameter < 3 microns
  - Length < 80 microns
  - Length to Diameter Ratio ≥ 3

5. DATA SOURCES

A brief overview of the respective data sources for the present study is shown in Table 2. Associated test descriptions are presented in Table 3. The various tests consisted of only a burn phase for the graphite composite samples. A very limited amount of data reduction and analysis was also performed for sticky paper records from NWC 12 in which spoiler residue material from NWC 11 was subjected to a burn phase within a pool fire for several minutes followed by an explosion of an high explosive charge in close proximity to the test sample.

The NWC 11, 12 and 13 tests were conducted in May 1978 at the Naval Weapon Center at China Lake with composite samples suspended several feet above 15.2m(50 ft.) JP-5 pool fires. The sticky paper records were approximately 20 X 25 cm and were located on wooden supports about 0.3m above ground. Tests BT-244, AF-4 and AF-6 were performed at the Naval Surface Weapons Center/Dahlgren Laboratory test chamber utilizing propane burners as the heat source. A 30 knot air flow source was initiated immediately following termination of a 20 minute burn phase for tests AF-4 and AF-6 in order to evaluate potential wind effects during an accidental aircraft fire. Tests D-1, D-2 and D-3 at the Dugway Proving Ground during October - November 1979 were similar to the NWC tests in that composite structural elements were mounted above a 10.7m (35 ft.) JP-4 pool fire, with the exception that NWC burn duration was of the order of several minutes whereas the DPG pool fires extended over a period of 20 minutes.

Table 2. Data Sources

RECORD	TEST NWC 11	TEST NWC 13	TEST BT–244	TEST AF–4	TEST AF–6	TEST D–1	TEST D–2	TEST D–3
TYPE	STICKY PAPER	STICKY PAPER	STICKY PAPER	STICKY PAPER	STICKY PAPER	PETRI DISH	PETRI DISH	PETRI DISH
NUMBER	10	10	3	6	6	3	6	18
LOCATION	140 N, 60 W 160 N, 60 W 160 N, 20 E 160 N, 60 E 170 N, 50 E 180 N, 60 W 210 N, 30 E 220 N, 20 W 250 N, 50 E 260 N, 60 E	140 N, 60 W 140 N, 0 E/W 150 N, 10 W 160 N, 40 W 180 N, 40 W 180 N, 20 W 180 N, 20 E 190 N, 50 W 190 N, 10 E 240 N, 0 E/W	NSWC TEST CHAMBER STATIONS 21,361 21,383 21, 412	NSWC TEST CHAMBER STATIONS 6 8 12 13 33 34	NSWC TEST CHAMBER STATIONS 5 11 21 24 30 42	RANGE 103 – 116M STATIONS 49 50 51	RANGE 103 – 116M STATIONS 26 27 28 29 30 31	RANGE 103 – 116M STATIONS 35 TO 52
PARTICLES	497	529	197	476	571	218	208	111

Table 3. Test Description

TEST DESIGNATION	TEST SAMPLE	COMPOSITE TYPE	TEST CONFIGURATION	BURN DURATION (MINUTES)
NWC 11	SPOILER	T300/5209	JP-5 POOL FIRE	4
NWC 13	COCKPIT	T300/5208	JP-5 POOL FIRE	6
BT-244	SPOILER	T300/5209	PROPANE BURNER	20
AF-4	PLATE 0.32 CM (1/8 ")	T300/5208	PROPANE BURN PLUS 30KT AIR FLOW	20 10
AF-6	PLATE 0.64 CM (1/4 ")	T300/5208	PROPANE BURN PLUS 30KT AIR FLOW	20 10
D-1 D-2 D-3	VARIOUS STRUCTURAL ELEMENTS	T300/5208	JP-4 POOL FIRE	20

6. ANALYTICAL RESULTS

During the course of data reduction of the NWC records, particles were observed which manifested a splitting or fibrillation process in that single fibers appeared to be segmented longitudinally along the axis of the fiber into slender individual fiber elements or essentially fibrils. Examples of particles of this nature are shown in Figures 1 and 2 for the NWC 11 records. Similar cases were also noted for NWC 12 and NWC 13 records. Some of the fibrillated particles do not consist of many fibrils whereas others reflect a substantial degree of splitting. In addition to longitudinal splitting there were also a number of particles which were also characterized by considerable fragmentation of the slender fibrils into short segments. Representative samples of fibrillated fibers with high micron particle density are shown in Figure 3. An enlarged view of one of the high micron particle density regions is presented in Figure 4.

It is readily apparent that the fibrillation effect constitutes a major source for the micron size domain of interest in consideration of potential health hazards. An effort was made to separate some of the fibrillated particles from the adhesive on the record into order to permit a somewhat detailed examination by means of a scanning electron microscope. However, a difficulty developed in preparing the fiber sample for SEM photography in that it was not possible to remove the adhesive completely without a significant layer adhering to the fiber surface. This layer was significantly greater than the depth of focus of the SEM equipment, and therefore, the particle could not be studied.

During this time period a series of large pool fire tests were being planned by NASA at the Dugway Proving Ground. It was recommended by TRW that Petri dish samplers be included in the instrumentation array in order to collect free particles deposited from the carbon fiber plume as it propagates downwind from the pool fire area. The carbon fibers collected in this manner were examined for any evidence of fibrillation effects. Results of the evaluation are described in Section 8.

The data reduction of the sticky paper records consisted essentially of determining the dimensions of the carbon fibers by means of optical microscopy at magnifications ranging from 50X to 1000X. For the case of the NWC records location of isolated particles with diameters of the order of 1 to 3 microns and lengths less than 100 microns was extremely difficult and time - consuming since the frequency of occurrence was quite small. It is quite probable that particles of this size with very low settling velocities would be propagated to significant distances downrange before being deposited on the ground. The NWC test records were only about 45 to 75 meters downrange from the pool fire.

The NWC data developed in the present study for micron size carbon fibers were predicated principally on the particles of this nature contained within or located in close proximity to the clusters associated with fibrillated fibers. In cases of high micron particle density regions as shown in Figures 3 and 4 only a representative number of particles were measured. In general it is estimated that the data reduction of micron size fibers was relatively incomplete and that the total quantities generated might warrant enhancement by perhaps a factor of 50 percent.

GAGE LOCATION 50N, 80W

GAGE LOCATION 50N, 80W

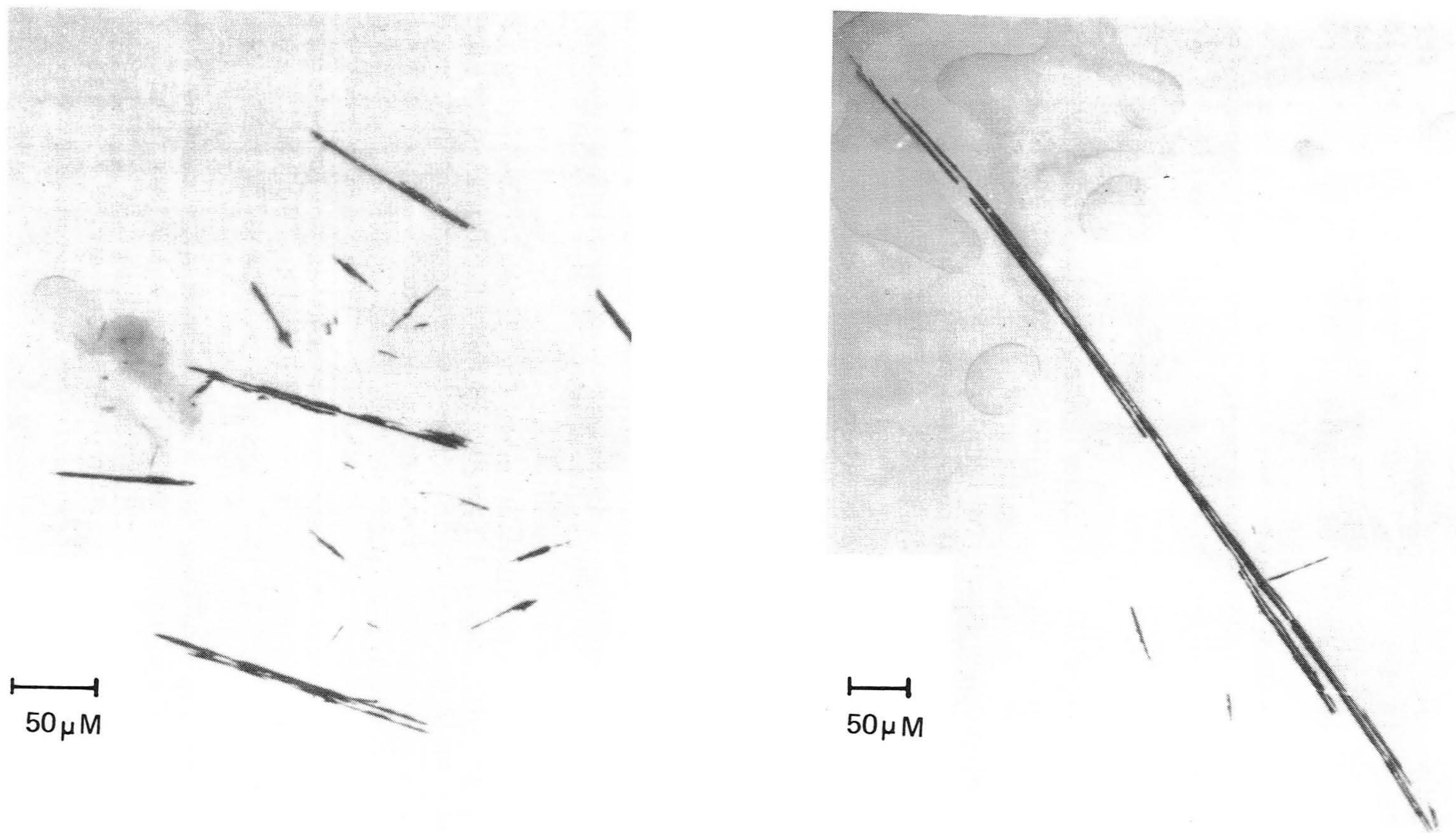


Figure 1. T300 Fibrillation Effect During NWC Spoiler Test 11 (I)

GAGE LOCATION 30N, 80W

GAGE LOCATION 50N, 0E/W

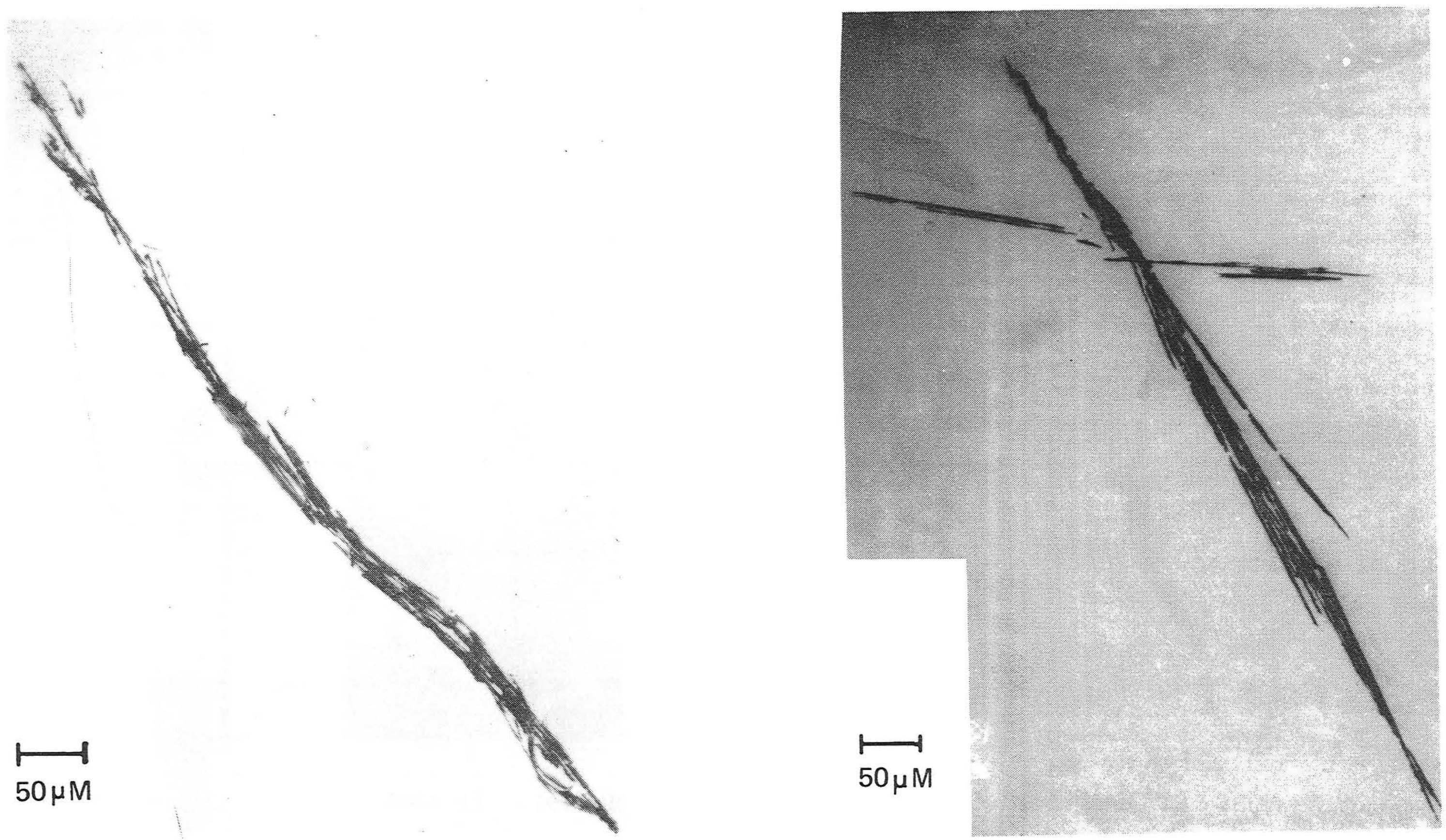


Figure 2. T300 Fibrillation Effect During NWC Spoiler Test 11 (II)

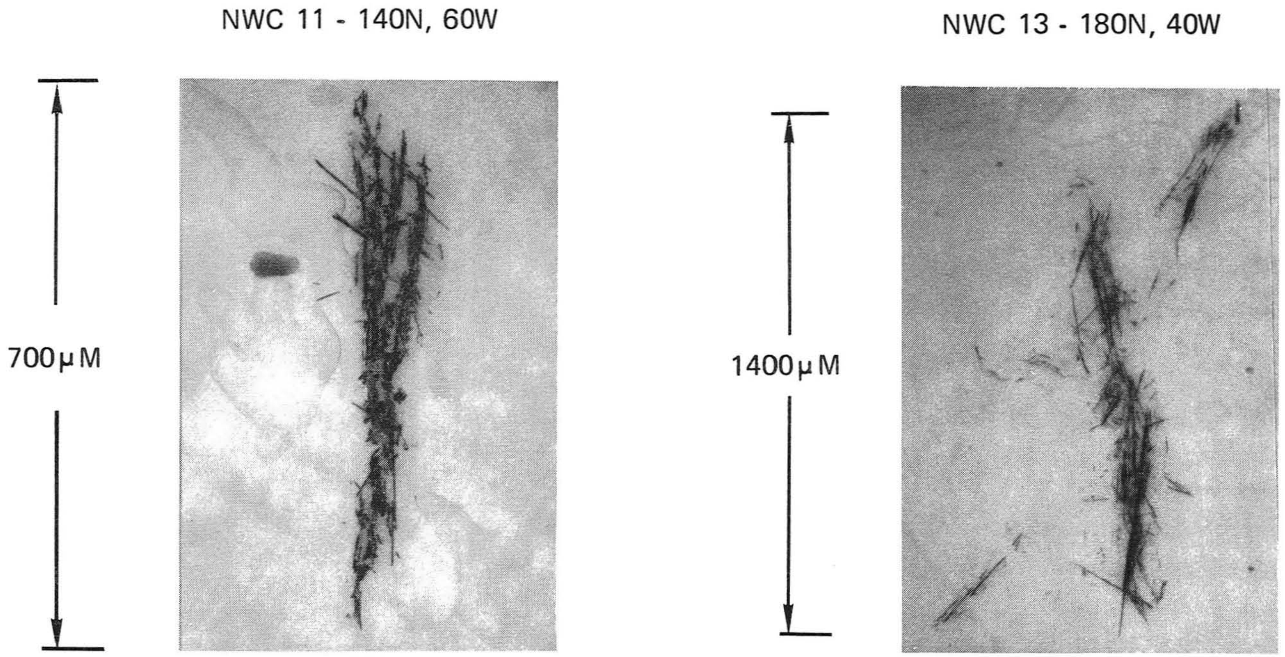


Figure 3. Representative Fibrillated Fibers With High Micron Particle Density

NWC 13 - 180N, 40W

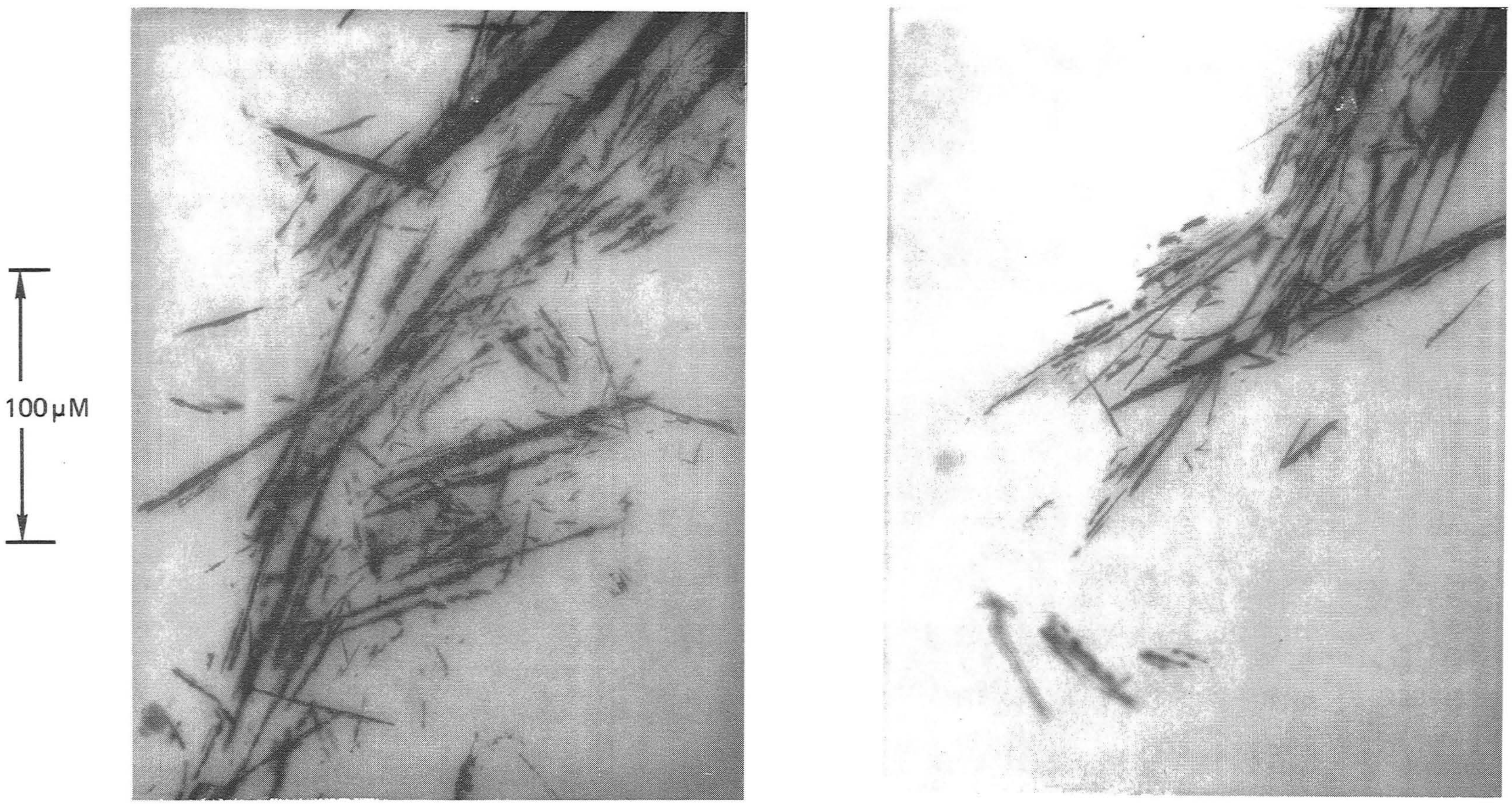


Figure 4. Enlarged View Of High Micron Particle Density Region

A tabulation of length versus diameter for all particles measured on NWC 11 records is shown in Table 4. Similar data for NWC 12 and NWC 13 records are given in Tables 5 and 6. A comparative evaluation is presented in Section 7. The respective data reduction areas were 700, 100 and 100 cm^2 for NWC 11, 12 and 13.

Length versus diameter correlations for the air flow tests AF-4 and AF-6 are shown in Tables 7 and 8, and for the spoiler test BT-244 in Table 9. It is noted that in the case of the air flow tests a considerable number of micron size particles were generated whereas none were observed for the spoiler test. This aspect is attributed to the enhancement of oxidation of the carbon fibers by the air flow as evidenced by the substantial glowing of the test samples after completion of the burn phase and upon activation of the air flow source.

Table 4. Spoiler Test NWC 11 - Length vs Diameter Correlation

DIAMETER INTERVAL (MICRONS)	LENGTH INTERVAL (MICRONS)												TOTAL	PERCENT
	5-10	10-20	20-40	40-75	75-150	150-300	300-600	600-1000	1000-2000	2000-4000	4000-8000	8000-16,000		
0-1	12	17	8	2	4	–	–	–	–	–	–	–	43	8.7
1-2	6	18	33	17	8	3	1	1	–	–	–	–	87	17.5
2-3	–	21	22	28	23	14	7	3	2	1	1	1	122	24.5
3-4	2	5	5	4	11	10	10	2	5	2	1	–	57	11.5
4-5	2	5	3	–	9	7	6	5	3	4	1	–	45	9.1
5-6	–	1	1	2	6	5	5	2	4	–	–	–	26	5.2
6-7	–	2	3	6	6	4	9	5	4	–	–	–	39	7.8
7-8	–	1	1	4	3	–	4	2	2	1	–	–	18	3.6
>8	–	1	15	23	5	1	4	7	2	1	1	–	60	12.1
TOTAL	22	71	91	86	75	43	46	27	22	9	4	1	497	100.0
PERCENT	4.4	14.3	18.3	17.3	15.1	8.7	9.3	5.4	4.4	1.8	0.8	0.2	100	

Table 5. Spoiler Test NWC 12 - Length vs Diameter Correlation

DIAMETER INTERVAL (MICRONS)	LENGTH INTERVAL (MICRONS)												TOTAL	PERCENT
	5–10	10–20	20–40	40–75	75–150	150–300	300–600	600–1000	1000–2000	2000–4000	4000–8000	8000–16,000		
0–1	–	–	3	1	1	1	–	–	–	–	–	–	6	8.1
1–2	1	1	3	1	–	1	3	–	–	1	–	–	11	14.9
2–3	–	3	–	6	1	3	2	–	2	–	–	–	17	23.0
3–4	–	–	1	–	–	2	4	1	–	–	–	–	8	10.8
4–5	–	–	–	1	–	–	2	1	2	1	–	–	7	9.4
5–6	–	–	1	–	1	–	2	–	–	1	1	–	6	8.1
6–7	–	–	2	1	–	2	–	–	–	–	–	–	5	6.8
7–8	–	–	1	2	–	1	–	–	1	–	1	–	6	8.1
>8	–	–	2	2	1	–	1	1	1	–	–	–	8	10.8
TOTAL	1	4	13	14	4	10	14	3	6	3	2	–	74	100
PERCENT	1.4	5.4	17.6	18.9	5.4	13.5	18.9	4.1	8.1	4.0	2.7	–	100	

Table 6. Cockpit Test NWC 13 - Length vs Diameter Correlation

DIAMETER INTERVAL (MICRONS)	LENGTH INTERVAL (MICRONS)												TOTAL	PERCENT
	5–10	10–20	20–40	40–75	75–150	150–300	300–600	600–1000	1000–2000	2000–4000	4000–8000	8000–16,000		
0–1	14	16	18	6	1	–	–	–	–	1	–	–	56	10.6
1–2	11	30	57	36	30	11	2	2	–	–	–	–	179	33.8
2–3	–	7	31	27	32	14	7	4	10	3	–	–	135	25.5
3–4	–	1	5	9	5	14	7	5	7	–	2	1	56	10.6
4–5	–	–	1	3	4	7	6	1	3	–	–	1	26	4.9
5–6	–	–	–	–	7	2	2	1	1	–	–	–	13	2.5
6–7	–	–	–	–	5	3	2	2	1	–	–	–	13	2.5
7–8	–	–	–	2	4	4	3	1	–	–	–	–	14	2.6
>8	–	–	–	1	5	8	5	6	8	4	–	–	37	7.0
TOTAL	25	54	112	84	93	63	34	22	30	8	2	2	529	100.0
PERCENT	4.7	10.2	21.2	15.9	17.6	11.9	6.4	4.1	5.7	1.5	0.4	0.4	100	

Table 7. Plate Test AF-4 - Length vs Diameter Correlation

DIAMETER INTERVAL (MICRONS)	LENGTH INTERVAL (MICRONS)												TOTAL	PERCENT
	5-10	10-20	20-40	40-75	75-150	150-300	300-600	600-1000	1000-2000	2000-4000	4000-8000	8000-16,000		
0-1	1	–	3	2	3	–	–	–	–	–	–	–	9	1.9
1-2	1	12	22	23	24	17	13	5	4	2	–	–	123	25.8
2-3	–	–	12	24	40	52	36	13	11	3	–	–	191	40.1
3-4	–	1	1	2	15	21	19	11	6	7	1	–	84	17.7
4-5	–	–	–	–	3	5	7	1	2	1	–	–	19	4.0
5-6	–	–	2	1	3	3	5	2	2	2	–	–	20	4.2
6-7	–	–	–	2	1	3	1	1	1	–	–	–	9	1.9
7-8	–	–	1	–	4	–	4	–	–	–	–	–	9	1.9
>8	–	–	–	2	4	1	2	3	–	–	–	–	12	2.5
TOTAL	2	13	41	56	97	102	87	36	26	15	1	–	476	100.0
PERCENT	0.4	2.7	8.6	11.8	20.4	21.4	18.3	7.6	5.5	3.1	0.2	–	100	

Table 8. Plate Test AF-6 - Length vs Diameter Correlation

DIAMETER INTERVAL (MICRONS)	LENGTH INTERVAL (MICRONS) 5–10	10–20	20–40	40–75	75–150	150–300	300–600	600–1000	1000–2000	2000–4000	4000–8000	8000–16,000	TOTAL	PERCENT
0–1	–	–	1	–	2	–	–	–	–	–	–	–	3	0.5
1–2	2	5	22	19	41	26	7	1	2	–	–	–	125	21.9
2–3	–	–	6	16	58	80	55	20	15	2	–	–	252	44.1
3–4	–	–	–	3	17	38	36	19	9	3	–	–	125	21.9
4–5	–	–	–	–	6	8	8	4	4	–	–	1	31	5.4
5–6	–	–	1	1	1	–	3	2	–	–	–	–	8	1.4
6–7	–	–	–	–	6	1	2	1	1	1	–	–	12	2.1
7–8	–	–	1	1	2	1	–	–	–	–	–	–	5	0.9
>8	–	–	–	1	–	4	1	2	1	1	–	–	10	1.8
TOTAL	2	5	31	41	133	158	112	49	32	7	–	1	571	100.0
PERCENT	0.3	0.9	5.4	7.2	23.3	27.7	19.6	8.6	5.6	1.2	–	0.2	100	

Table 9. Spoiler Test BT-244 - Length vs Diameter Correlation

DIAMETER INTERVAL (MICRONS)	LENGTH INTERVAL (MICRONS)												TOTAL	PERCENT
	5-10	10-20	20-40	40-75	75-150	150-300	300-600	600-1000	1000-2000	2000-4000	4000-8000	8000-16,000		
0-1	–	–	–	–	–	–	–	–	–	–	–	–	–	–
1-2	–	–	–	–	–	–	–	–	–	–	–	–	–	–
2-3	–	–	–	–	–	–	–	–	–	–	–	–	–	–
3-4	–	–	–	–	–	1	1	–	1	–	–	–	3	1.5
4-5	–	–	–	1	6	3	–	1	–	1	–	–	12	6.1
5-6	–	–	2	5	22	13	5	4	3	2	–	–	56	28.4
6-7	–	–	3	17	18	10	7	3	1	–	–	–	59	30.0
7-8	–	–	–	6	8	8	4	4	–	–	–	–	30	15.2
>8	–	–	1	12	11	9	4	–	–	–	–	–	37	18.8
TOTAL	–	–	6	41	65	44	21	12	5	3	–	–	197	100.0
PERCENT	–	–	3.1	20.8	33.0	22.3	10.7	6.1	2.5	1.5	–	–	100	

7. COMPARATIVE EVALUATION

TEST DATA OVERVIEW

Comparisons of respective diameter and length distributions for NWC 11 and NWC 12 are shown in Figure 5. The diameter distributions are quite similar. In the case of the length comparison the NWC 11 data reflect a somewhat higher frequency of occurrence for the shorter fiber lengths. The average length for the NWC 11 distribution is 315 microns whereas the value for the NWC 12 data is 544 microns.

Figure 6 presents a set of comparisons of the diameter and length distributions for NWC 11 and NWC 13. In general the respective distributions are quite similar. The average length for the NWC 13 data is 307 microns which compares favorably with the value of 315 microns for NWC 11 indicated above.

A comparison of single fiber distributions for the spoiler Tests NWC 11 (pool fire) and BT-244 (propane burn) is shown in Figure 7. A substantial reduction in fiber diameter due to oxidation is readily apparent for the NWC 11 data whereas relatively insignificant diameter reduction is manifest by the BT-244 results. In the case of the length spectrum comparison the NWC 11 distribution indicates a higher frequency for smaller lengths than for the BT-244 data. A possible explanation for the gross difference in diameter reduction is that for the propane burn a non-oxidizing environment envelopes the test sample.

For the case of the air flow tests AF-4 and AF-6 a substantial reduction in diameter occurred from the initial diameter value of about 7 microns as noted in Figure 8. The length distributions for the two tests appear to be quite comparable. The test sample for AF-4 consisted of an 1/8" plate of T-300/5208 composite which was rated as resin-rich in comparison to the 1/4" plate of T-300/5208 composite for test AF-6 which was evaluated as resin-poor due to some deficiency in the fabrication process. It was initially anticipated that the respective fiber distributions would reflect some variations due to the thickness and resin content differences. However, the comparisons of Figure 8 indicate that the relative frequencies of occurrence over the various diameter and length intervals are quite similar. The comparison of areal densities in Table 10 shows that the density distributions were quite uniform over the entire test area for both tests. Table 11 indicates that for AF-4 the relative occurrence of single fiber diameters less than 3 microns was 77% as compared to 79% for AF-6. It appears reasonable to conclude that the test conditions were insufficient to permit differentiation between the two types of test samples.

The Petri dish samplers at the DPG tests were mounted horizontally at the top of wooden stakes about 2 feet above the ground. Located in close proximity on each stake were bridal veil gages (4" diameter, stainless steel 1 mm mesh, adhesive coating) oriented with axis parallel to the ground pointing toward ground zero. At the base of each stake a sticky paper record was located lying on the ground. The bridal veil records were intended to establish the horizontal component of the fiber pattern and the sticky paper records to determine the corresponding vertical deposition component at the same location.

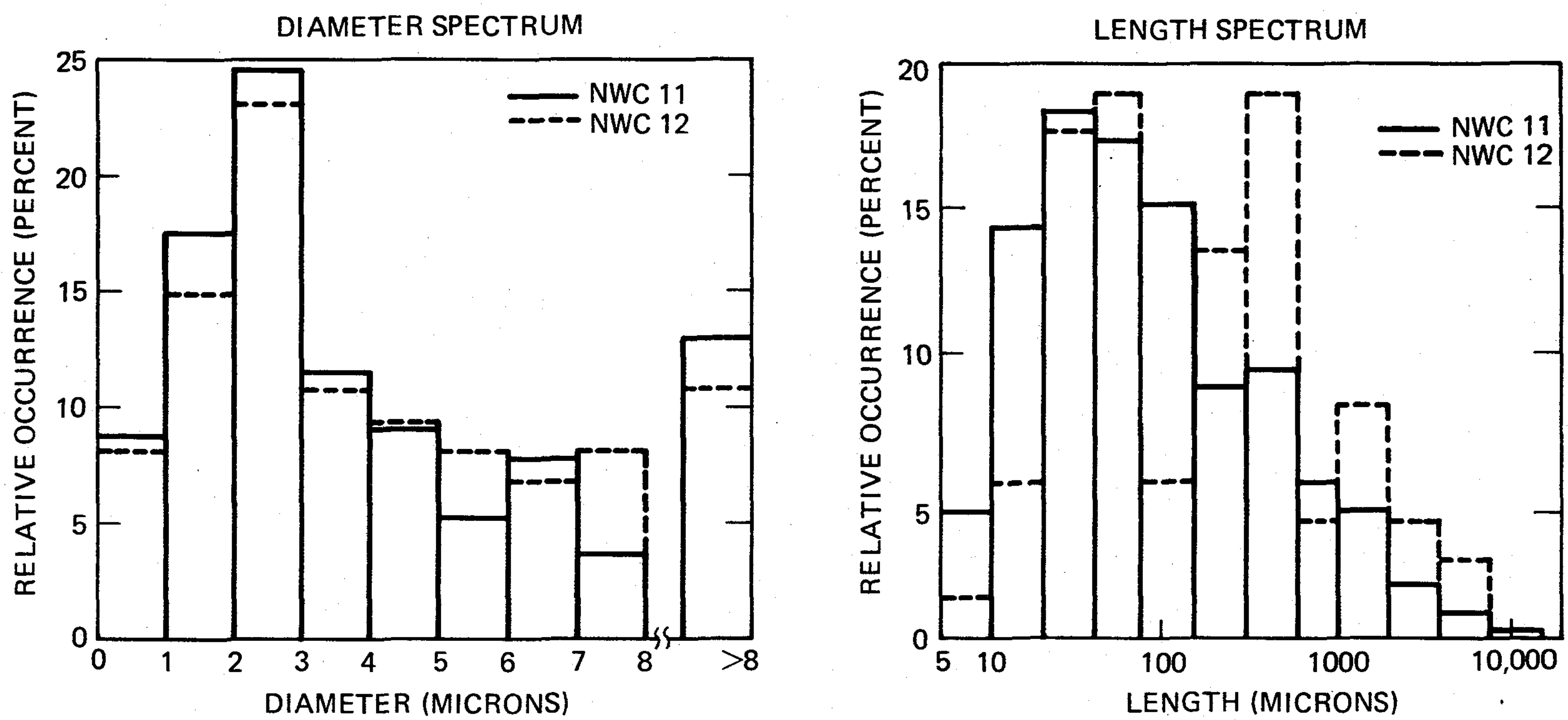


Figure 5. Comparison of NWC 11 and NWC 12 Distributions

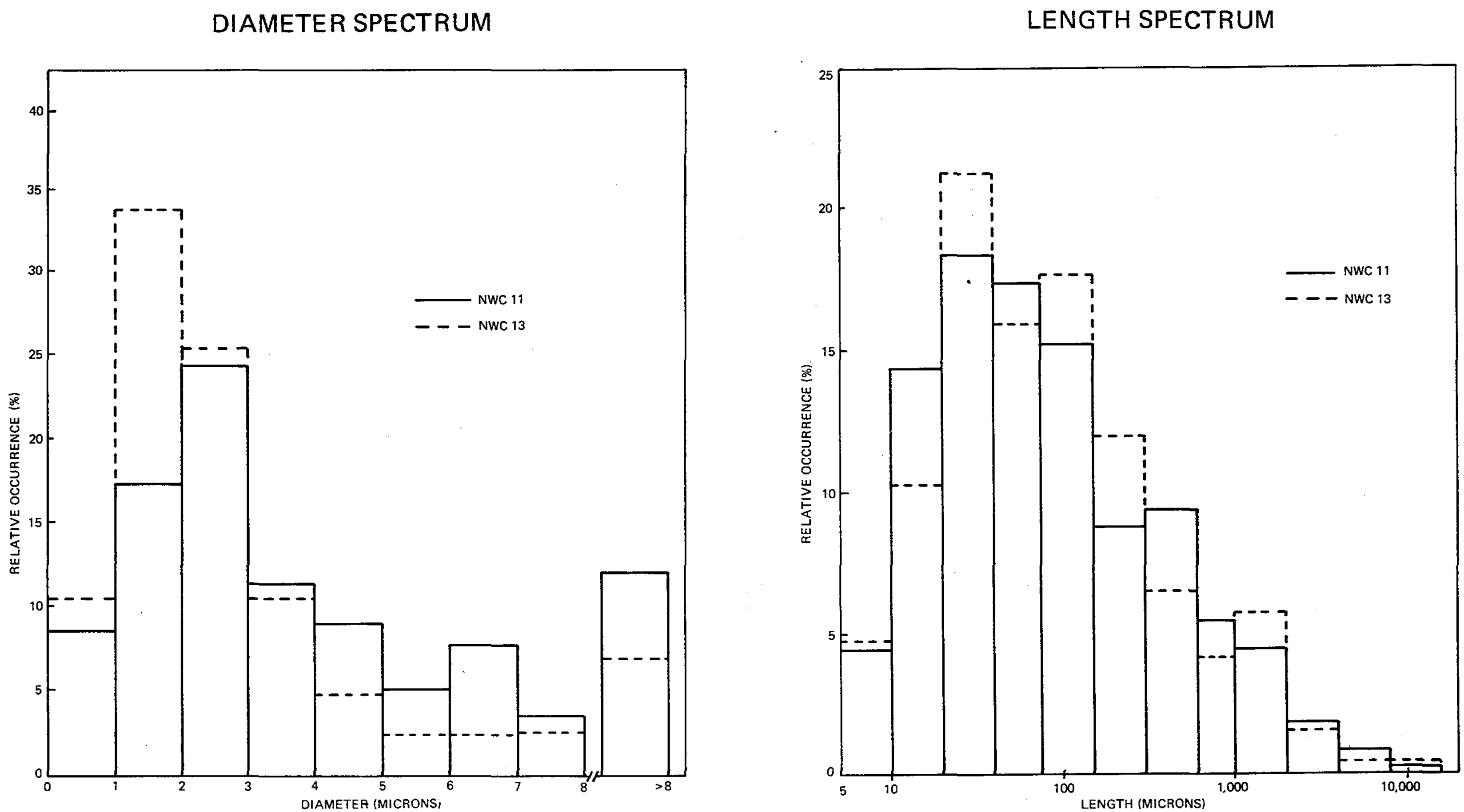


Figure 6. Comparison of NWC 11 & NWC 13 Distributions

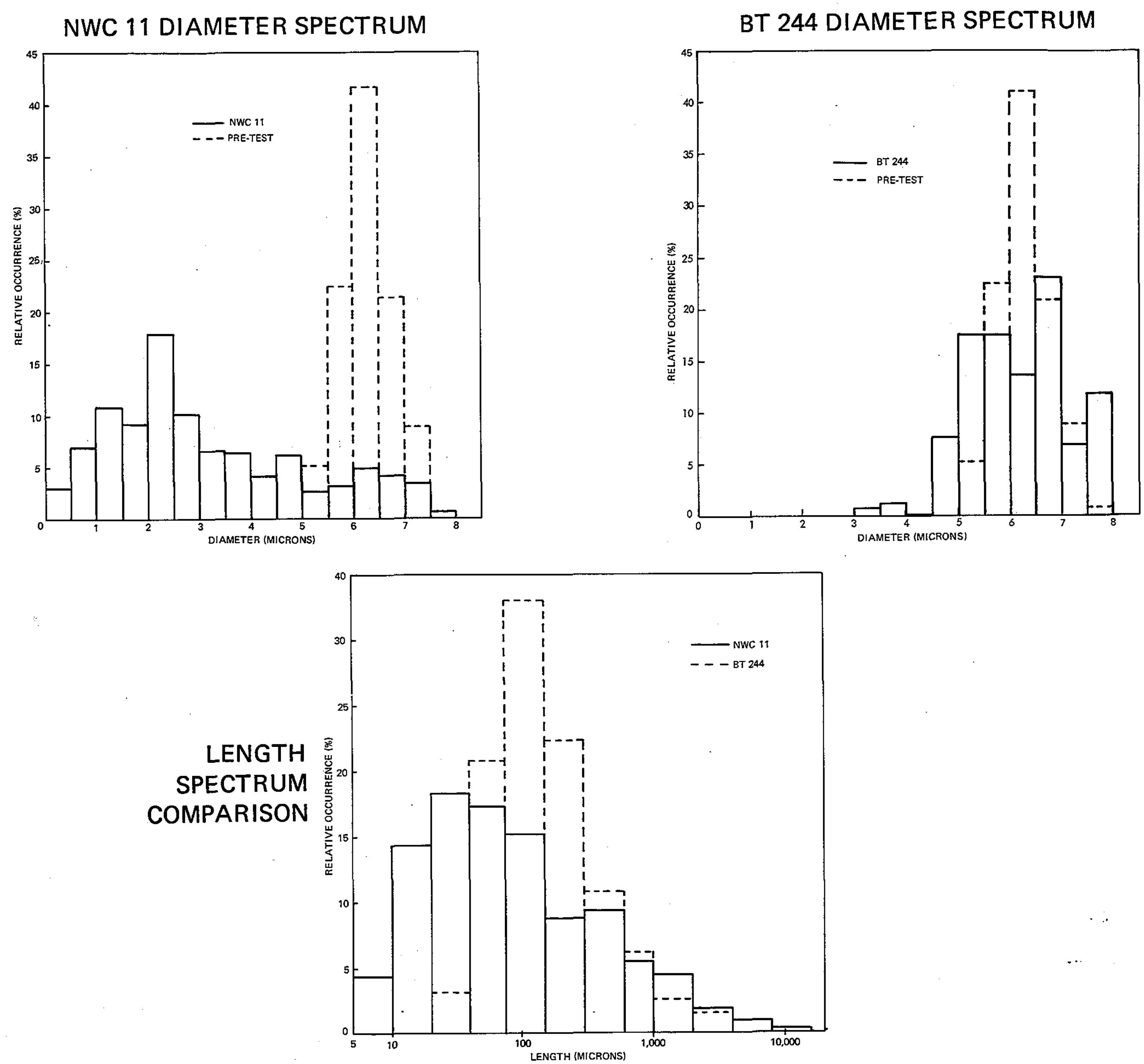


Figure 7. Single Fiber Distributions for Spoiler Test Sample

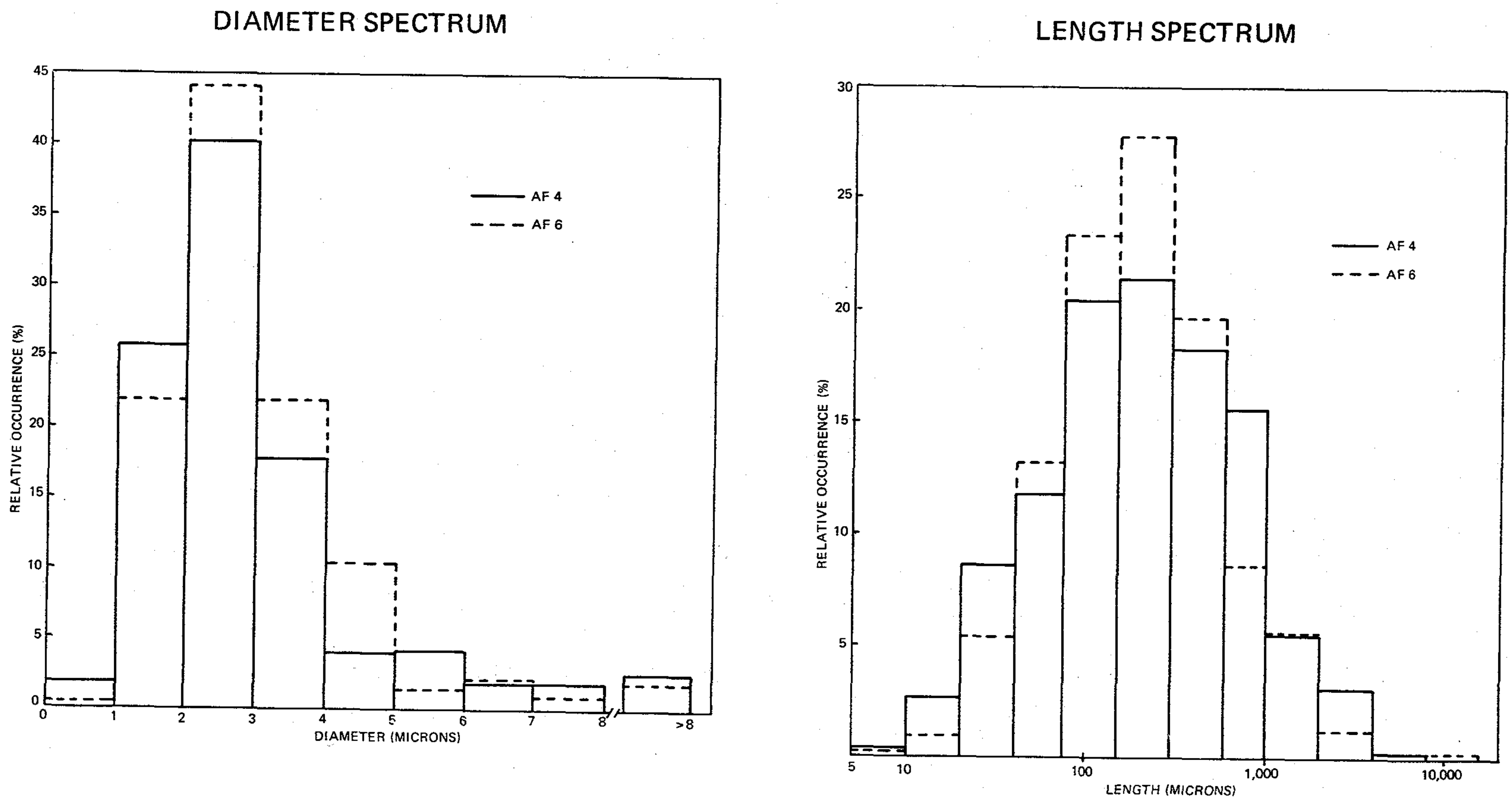


Figure 8. Comparison of AF-4 & AF-6 Distributions

Table 10. Areal Density Comparison for Air Flow Tests

TEST DESIGNATION	TEST SAMPLE	RECORD NUMBER	DATA REDUCTION AREA (CM^2)	PARTICLE NUMBER	A REAL DENSITY (F/M^2)
AF–4	PLATE 0.32 CM (1/8") (T300/5208)	6	4	109	27,200
		8	4	82	20,500
		12	4	124	31,000
		13	3	64	21,300
		33	2	35	17,500
		34	2	62	31,000
			19 TOTAL	476 TOTAL	24,800 AVERAGE
AF–6	PLATE 0.64 CM (1/4") (T300/5208)	5	4	77	19,200
		11	4	95	23,800
		21	4	103	25,800
		24	4	108	27,000
		30	4	115	28,800
		42	4	73	18,200
			24 TOTAL	571 TOTAL	23,800 AVERAGE

Table 11. Micron Domain Comparison for Tests AF-4 and AF-6

DIAMETER INTERVAL (MICRONS)	TEST AF-4 RELATIVE OCCURRENCE (%)	TEST AF-6 RELATIVE OCCURRENCE (%)	LENGTH INTERVAL (MICRONS)	TEST AF-4 RELATIVE OCCURRENCE (%)	TEST AF-6 RELATIVE OCCURRENCE (%)
0–0.5	1.9	0.5	0–5	–––	–––
0.5–1.0	12.2	12.6	5–10	0.4	0.3
1.0–1.5	13.7	9.3	10–20	2.7	0.9
1.5–2.0	23.7	21.7	20–40	8.6	5.4
2.0–2.5	16.4	22.4	40–75	11.8	7.2
2.5–3.0	9.5	12.6			
TOTAL	77.4	79.1	TOTAL	23.5	13.8

A correlation between areal densities for the TRW Petri dish data and the DPG bridal veil and sticky paper data for tests D-1, D-2 and D-3 is shown in Table 12. The DPG results are based on preliminary data by John Trethewey of the Dugway Proving Ground. The composite mass for the three respective tests were approximately 45, 45 and 74 kg. Wind velocities for the respective tests were 6.4, 5.8 and 5.3 meters per second. Estimates of fiber exposure levels may be determined by either of two methods, namely:

$$E = \frac{\rho_{BV}}{V_w} \quad \text{or} \quad E = \frac{\rho_{PD}}{V_D} = \frac{\rho_{SP}}{V_D}$$

where P_{BV}, P_{PD} and P_{SP} are the areal densities for the bridal veil Petri dish and sticky paper records, and V_W and V_D are the wind and deposition velocities.

It appears reasonable to assume that the values of the ratio V_w/V_D are quite comparable for each of the three tests. Therefore, it would be anticipated that the test data would reflect an equivalence of the areal density ratios namely:

$$\frac{\rho_{BV}}{\rho_{PD}} = \frac{\rho_{BV}}{\rho_{SP}}$$

This relation appears to be reasonably satisfied by the average values for test D-3 shown in Figure 12. However, for the case of test D-2, the two ratios differ by a factor of about 5. It appears, therefore, that the sticky paper deposition density was about 5 times greater than the Petri dish density for test D-2, but relatively equivalent for test D-3. No comparison can be made for test D-1 since no sticky paper data were available.

The comparison of single fiber diameter distributions in Table 13 indicates that the diameter spectrum of tests NWC 11, NWC 13, AF-4 and AF-6 are quite similar, with the data of BT-244 alone reflecting essentially no loss in fiber diameter. Table 14 presents a similar comparison of single fiber length distributions, indicating again a similarity between tests NWC 11, NWC 13, AF-4 and AF-6, with surprisingly little evidence of fibers of lengths less than 75 microns for the three DPG tests.

The comparison of relative fiber frequencies shown in Table 15 indicates that the occurrence of single fibers with lengths greater than or equal to 1 mm average about 6% for all of the tests and about 85% for lengths less than 1 mm. For the case of micron fibers in the range of diameters less than 3 microns and lengths less than 80 microns the NWC average is a factor of about 2.4 greater than the result for the air flow tests, with no particles in this domain observed for BT-244.

MICRON FIBER DOMAIN

An estimate of the occurrence frequency of fibrillated fibers for NWC 11 is shown in Figure 9 to be 17.7%, with an average of 7.5 particles incorporated in the data reduction per fibrillated fiber. Corresponding values for NWC 13 are given in Table 16 as 23.0% for the frequency of fibrillated fibers and an average of 8.7 particles per fibrillated fiber. It appears, therefore, that about 20% of the fiber elements evaluated during the data reduction process were of the fibrillated type, with an average of about 8 particles per fibrillated fiber. It was noted earlier that the data reduction of the fibrillated fibers was incomplete and that an enhancement factor of about 50 percent might be warranted.

A comparison of areal densities for NWC 11 and NWC 12 is shown in Table 17.

Table 12. Correlation Between TRW Petri Dish Data and DPG Bridal Veil and Sticky Paper Data

TEST D-1[1]				TEST D-2					TEST D-3				
LOCATION	PD FIBER NUMBER	PD AREAL DENSITY (F/M^2)	ρ_{BV}/ρ_{PD}	LOCATION	PD FIBER NUMBER	PD AREAL DENSITY (F/M^2)	ρ_{BV}/ρ_{PD}	ρ_{BV}/ρ_{SP}	LOCATION	PD FIBER NUMBER	PD AREAL DENSITY (F/M^2)	ρ_{BV}/ρ_{PD}	ρ_{BV}/ρ_{SP}
49	64	11,300	4.9	26	21	3,700	6.3	56.1	35	3	529	30.3	14.1
50	85	15,000	4.5	27	9	1,100	59.2[2]	70.2	36	3	529	118.3[2]	46.6
51	69	12,200	5.1	28	34	6,000	15.1	41.6	37	8	1,410	30.8	28.1
	218 TOTAL		4.8 AVERAGE	29	56	9,880	7.3	27.9	38	4	705	81.2	61.5
				30	30	5,290	8.5	29.1	39	3	529	63.5	162.2[2]
				31	58	10,200	4.8	22.6	41	4	705	45.5	34.5
					208 TOTAL		8.4 AVERAGE	41.2 AVERAGE	42	6	1,060	40.4	46.0
									43	1	176	212.5[2]	10.3
									45	8	1,410	28.1	64.0
									46	15	2,650	10.7	39.0
									48	6	1,060	28.0	48.0
									50	4	6,490	9.2	63.0
									51	5	11,800	13.4	38.2
									52	5	2,670	3.0	12.9
										111 TOTAL		32.0 AVERAGE	38.9 AVERAGE

(1) NO STICKY PAPER DATA AVAILABLE
(2) EXCLUDED FROM AVERAGE

Table 13. Comparison of Single Fiber Diameter Distributions

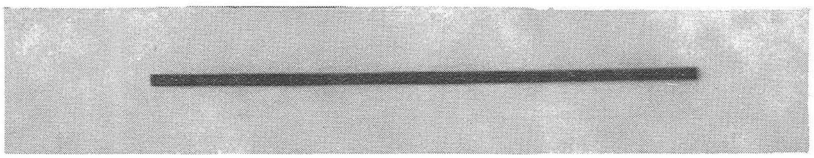
DIAMETER INTERVAL (MICRONS)	TEST NWC 11 (%)	TEST NWC 13 (%)	TEST AF-4 (%)	TEST AF-6 (%)	TEST BT-244 (%)
0–1	9.8	11.4	1.9	0.5	–
1–2	19.9	36.4	26.5	22.3	–
2–3	27.9	27.4	41.2	44.9	–
3–4	13.1	11.4	18.2	22.3	1.9
4–5	10.3	5.3	4.1	5.5	7.5
5–6	6.0	2.6	4.3	1.4	35.0
6–7	8.9	2.6	2.0	2.2	36.8
7–8	4.1	2.9	1.9	0.9	18.8

Table 14. Comparison Of Single Fiber Length Distributions

LENGTH INTERVAL (MICRONS)	TEST NWC 11 (%)	TEST NWC 13 (%)	TEST BT-244 (%)	TEST AF-4 (%)	TEST AF-6 (%)	TEST D–1 (%)	TEST D–2 (%)	TEST D–3 (%)
5-10	5.1	5.1	–	0.4	0.4	–	–	–
10-20	16.0	11.0	–	2.8	0.9	–	–	–
20-40	17.4	22.3	3.1	8.8	5.5	–	–	–
40-75	14.4	16.9	18.1	11.7	7.1	0.9	–	0.9
75-150	16.0	17.9	33.8	20.1	23.7	3.7	8.2	17.1
150-300	9.6	11.1	21.9	21.8	27.4	24.3	17.3	27.1
300-600	9.6	5.9	10.6	18.3	19.8	25.7	27.4	26.1
600-1000	4.6	3.2	7.5	7.1	8.4	18.8	19.2	13.5
1000-2000	4.6	4.5	3.1	5.6	5.5	19.7	17.8	5.4
2000-4000	1.8	0.8	1.9	3.2	1.1	5.0	7.2	7.2
4000-8000	0.7	0.4	–	0.2	–	1.9	1.9	2.7
8000-16,000	0.2	0.4	–	–	0.2	–	1.0	–
TOTAL	100	100	100	100	100	100	100	100

Table 15. Relative Fiber Frequency Comparison

TEST	MULTIPLE FIBERS (D≥8μM) (%)	SINGLE FIBERS (D<8μM)		MICRON FIBERS D<3μM, L<80μM (%)
		L ≥ 1MM (%)	L < 1MM (%)	
NWC 11	12.1	6.4	81.5	38.4
NWC 13	7.0	5.7	87.3	49.5
AVERAGE	9.6	6.0	84.4	44.0
AF-4	2.5	8.8	88.7	22.7
AF-6	1.8	6.6	91.6	14.4
AVERAGE	2.2	7.7	90.1	18.6
BT-244	18.8	4.0	77.2	0

FIBER TYPE	FIBER NUMBER	PARTICLE NUMBER
FIBRILLATED 	41	306
NON-FIBRILLATED 	191	191
TOTAL	232	497

AVERAGE NUMBER OF PARTICLES PER FIBRILLATED FIBER $\frac{306}{41} = 7.5$

OCCURRENCE FREQUENCY OF FIBRILLATED FIBERS $\frac{41}{232} = 17.7\%$

Figure 9. Fibrillated Fiber Frequency for Test NWC 11

Table 16. Relative Occurrence Of Fibrillated Fibers

TEST	TOTAL PARTICLE NUMBER	PARTICLE SOURCE		FIBRILLATED[1] FIBERS		FIBRILLATED PLUS NON-FIBRILLATED FIBERS	PERCENT OF FIBRILLATED FIBERS
		NON-FIBRILLATED FIBERS	FIBRILLATED FIBERS	NUMBER	AVERAGE PARTICLES PER FIBER		
NWC 11	497	191	306	41	7.5[2]	232	17.7
NWC 13	529	147	382	44	8.7[2]	191	23.0
				AVERAGE	8.1[2]		20.4

(1) COMPOSITE GROUP FROM PARENT SINGLE FIBER PROPAGATED AS COLLECTIVE UNIT

(2) LOWER LIMIT DUE TO INCOMPLETE DATA REDUCTION OF FIBRILLATED FIBERS

Table 17. Comparison of Areal Densities

Test	Initial CF Mass (KG)	Data Reduction (CM^2)	Total Particle Number	Areal Density (F/M^2)
NWC 11	2.6	700	497	7100
NWC 12	1.3	100	74	7400

It is interesting to note that the areal densities are equivalent indicating that a particle enhancement by a factor of 2 occurred as a result of the burn/explode mode of NWC 12 compared to the burn only for NWC 11, and thus compensated for the mass difference of a factor of 2.

Development of the micron fiber criteria in the present study has been predicated principally on the data obtained from NWC 11 and 13. Table 18 presents an estimate of the relative occurrence of the micron fiber domain for these respective tests. The initial carbon fiber mass for NWC 13 was a factor of 6.2 greater than the initial CF mass for NWC 11. However, the corresponding ratio of areal densities is 7.4. The comparable values of mass ratio and areal density ratio indicates that the total particle deposition per unit mass is equivalent. Taking into account the relative similarity of the diameter and length distributions for NWC 11 and NWC 13 indicated in Figure 6, it appears reasonable to assume that a combination of the micron fiber data for these two tests is appropriate for the present criteria analysis.

The combined distribution for the micron fiber domain is shown in Table 19 for diameters less than 3 microns and lengths less than 80 microns. A correlation of the length to diameter ratios versus diameter is presented in Table 20. The lower limits were respectively 0.4 microns for diameter, 2 microns for length and 3 for length to diameter ratio. The lower limit of optical microscopy is estimated to be about 0.5 microns. Characterizations of the micron fiber frequency distributions over a spectrum of intervals of diameter, length and length to diameter ratio are plotted in Figure 10. An overview of the regions of relatively increasing micron fiber occurrence is presented in Figure 11.

With reference to Table 18 the average frequency of occurrence for NWC 11 and NWC 13 of single fibers in the micron fiber domain is 44% as compared to 6% for lengths less than or equal to 1 mm, or a ratio of 7.3. To account for incomplete data reduction of the fibrillated fibers it appears reasonable to increase this ratio to 10.

Table 18. Relative Occurrence Of Micron Fiber Domain

TEST	TEST SAMPLE	INITIAL CF MASS (KG)	DATA REDUCTION AREA (CM^2)	TOTAL PARTICLE NUMBER	PARTICLE AREAL DENSITY (PER M2)	SINGLE FIBERS			
						LENGTH $\geq$ 1MM		DIAMETER $< 3\mu M$, LENGTH $< 80\mu M$	
						NUMBER	PERCENT	NUMBER	PERCENT
NWC 11	SPOILER	2.6	700	497	7,100	32	6.4		38.4
NWC 13	COCKPIT	16.2	100	529	52,900	30	5.7	262	49.5
						AVERAGE	6.0		44.0

- EQUIVALENT PARTICLE DENSITIES PER UNIT MASS
- SINGLE FIBERS IN $L \geq 1$ MM DOMAIN — 6%
- SINGLE FIBERS IN $D < 3\mu M$, $L < 80\mu M$ DOMAIN — 44%
- MICRON FIBER FREQUENCY RATIO — 7.3
- MODIFIED RATIO DUE TO INCOMPLETE DATA REDUCTION OF FIBRILLATED FIBERS — 10

Table 19. Micron Fiber Domain - Length vs Diameter Correlation

DIAMETER INTERVAL (MICRONS)	LENGTH INTERVAL (MICRONS)									
	0–10 (1)	10–20	20–30	30–40	40–50	50–60	60–70	70–80	TOTAL	PERCENT
0–0.5 (2)	6	4	2	1	—	—	—	—	13	2.9
0.5–1.0	20	29	13	10	4	2	2	1	81	17.9
1.0–1.5	16	35	24	24	10	9	5	3	124	27.4
1.5–2.0	3	13	26	16	5	8	11	8	90	19.8
2.0–2.5	—	22	23	16	10	14	11	9	105	23.2
2.5–3.0	—	5	8	7	4	7	5	4	40	8.8
TOTAL	43	108	96	74	33	40	34	25	453	100.0
PERCENT	9.5	23.9	21.2	16.3	7.3	8.8	7.5	5.5	100	

(1) LOWER LENGTH LIMIT-2 MICRONS

(2) LOWER DIAMETER LIMIT-0.4 MICRONS

Table 20. Micron Fiber Domain - L/D Ratio vs Diameter Correlation

DIAMETER INTERVAL (MICRONS)	LENGTH TO DIAMETER RATIO									TOTAL	PERCENT
	0–10 (1)	10–20	20–30	30–40	40–50	50–60	60–70	70–80	>80		
0–0.5 (2)	1	4	1	3	1	1	1	1	–	13	2.9
0.5–1.0	9	21	10	18	7	5	4	2	5	81	17.9
1.0–1.5	17	39	20	26	11	8	–	3	–	124	27.4
1.5–2.0	13	33	15	16	11	2	–	–	–	90	19.8
2.0–2.5	26	38	16	25	–	–	–	–	–	105	23.2
2.5–3.0	11	17	1	11	–	–	–	–	–	40	8.8
TOTAL	77	152	63	99	30	16	5	6	5	453	100.0
PERCENT	17.0	336	13.9	21.8	6.6	3.6	1.1	1.3	1.1	100	

(1) LOWER L/D LIMIT - 3

(2) LOWER DIAMETER LIMIT-0.4 MICRONS

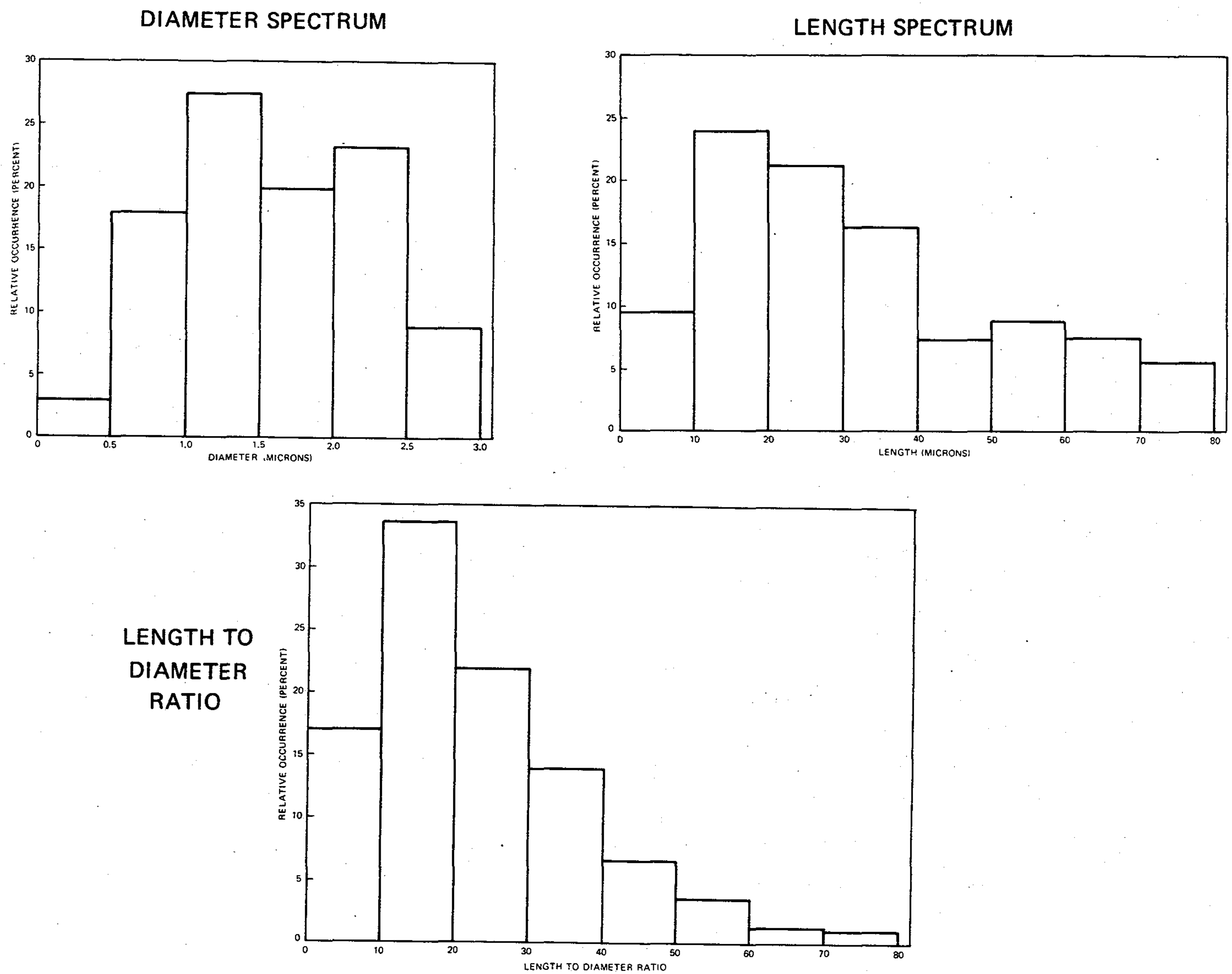


Figure 10. Micron Fiber Characteristics

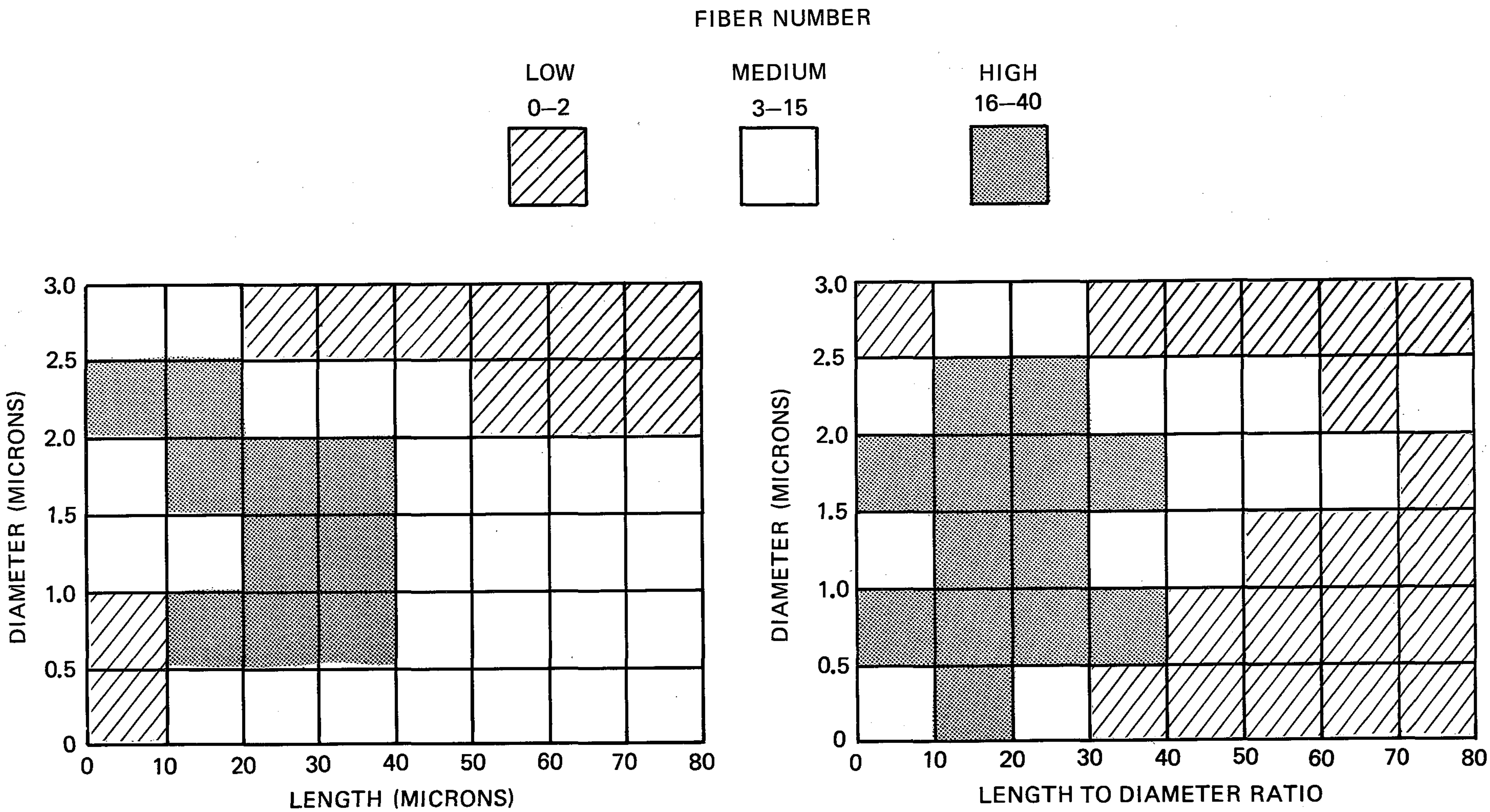


Figure 11. Relative Micron Fiber Distribution

8. FIBRILLATION PHENOMENA

BASIC CHARACTERISTICS

Since the fibrillated fibers constituted the major source of micron fiber data an effort was made to establish conclusively that the fibrillated particles originated from parent graphite fibers contained in the composite test sample rather than some type of foreign debris that had been drawn into the fire environment as a result of the associated air currents.

The approach taken was to attempt to correlate the levels of unique trace elements characteristic of T-300 carbon fibers with corresponding data from representative samples of fibrillated fibers, and establish some degree of equivalence. Various analytical techniques were given consideration, such as:

- SEM (Scanning Electron Microscope)
- EDAX (Energy Dispersive Analysis of X-Rays)
- IMMA (Ion Microprobe Mass Analyzer)
- EPB (Electron Probe Microanalyzer)
- ESCA (Electron Spectroscopy for Chemical Analysis)
- Auger Electron Microscope

Sample preparation consisted of separating a fibrillated fiber from the sticky paper adhesive by means of a laboratory solvent and mounting it on the surface of an Aluminum support block. For purposes of comparison, three additional samples were mounted adjacent to the fibrillated fiber of the following nature: (1) a single fiber of non-fibrillated appearance removed from the sticky paper record, (2) a portion of spoiler T-300 fibers obtained as residue of an acid test for resin content, and (3) a segment of raw T-300 yarn obtained from the Air Force Materials Laboratory.

Tests were conducted with the SEM, EDAX, IMMA and EPB. Inconsistent data on trace elements were obtained for the two T-300 fiber samples indicating the possibility of surface contamination anomalies. With reference to the fibers removed from the sticky paper, although readily photographed by means of optical microscopy, they were not "observed" by any of the respective electron microscopy techniques. It appears that the depths of focus in each case were insufficient to penetrate the surface layer of adhesive on the fibers that could not be removed by any type of available solvent.

As a result of this development it appeared advisable to obtain bulk measurements of trace elements by DC arc emission tests and to possibly utilize the capability of the IMMA equipment to penetrate sample depths by continuous sputtering of the surface over an extended period beyond the depth of the adhesive layer. This experiment has not been performed to date.

Another approach toward resolving the issue appeared to lend itself by means of introducing Petri dish samplers as part of the passive instrumentation array then being installed for the composite burning tests at Dugway Proving Ground. A TRW recommendation for the Petri dish supplement was made to NASA on the basis that in all probability fibrillated fibers would be generated during the DPG tests which were quite similar in some respects to the NWC tests, and that availability of free fibrillated fibers unencumbered by an adhesive layer would be very helpful toward the investigations of fibrillation effects of the present study.

The recommendation was approved by NASA and the Petri dish locations for tests D-1, D-2 and D-3 are shown in the instrumentation layout of Figure 12. The range from ground zero was 103-166 meters, with the respective wind directions for each test indicated by the arrows. It appeared that the Petri dish samplers were favorably located for the deposition of representative particulate from the burning composite (T-300/5208) test samples.

A total of about 500 particles were examined by means of optical microscopy similar to the techniques applied in data reduction of the NWC test records and no fibers were observed with significant fibrillation effects comparable to the types of NWC fibers of Figures 1 to 4. However, in several cases examinations with optical magnifications of about 50X to 100X indicated some degree of fiber separation at various positions along a single fiber.

These special cases were studied by means of SEM photography at magnifications of 1000X to 20,000X with the result that fibrillation effects became readily apparent. Examples of the different manifestations of the fibrillation phenomena are shown in Figures 13 and 14 with arbitrary categorization of the types of effects as singular area, hollow trunk, flaking, eye-of-the-needle and submicron fibers. These photographs appear to give striking evidence of the spectrum of oxidation effects caused by the fire environment leading to what has been designated as fibrillation phenomena and indicating conclusively a source of micron size fibers from a parent single carbon fiber.

It was of interest to study the pattern of separation of single fibrils of significant lengths. The result in one case is reflected by the sequence of photographs presented in Figure 15, each corresponding to 3000X magnification with the indicated vertical scale of 2 microns and horizontal scale of 50 microns applicable similarly to each photograph. The overall length covered was about 250 microns. Each photograph is essentially an extension at the left of the photograph immediately adjacent and directly below it with a small overlap for continuity identification.

Separation of the two fibrils in the uppermost photograph appear to continue over a length of about 100 microns until the advent of a third fibril in the middle photograph. Subsequently the three fibrils merge within a length of about 125 microns with evidence only of a single fine line of separation at the termination in the bottom photograph. At the far right of the bottom photograph a fiber is noted with an average diameter of about 1 micron and length of about 20 microns. However, over a length of about 3 microns at each end the fiber diameter is gradually reduced down to a value of a small fraction of a micron. This manifestation of diameter reduction at the ends is quite

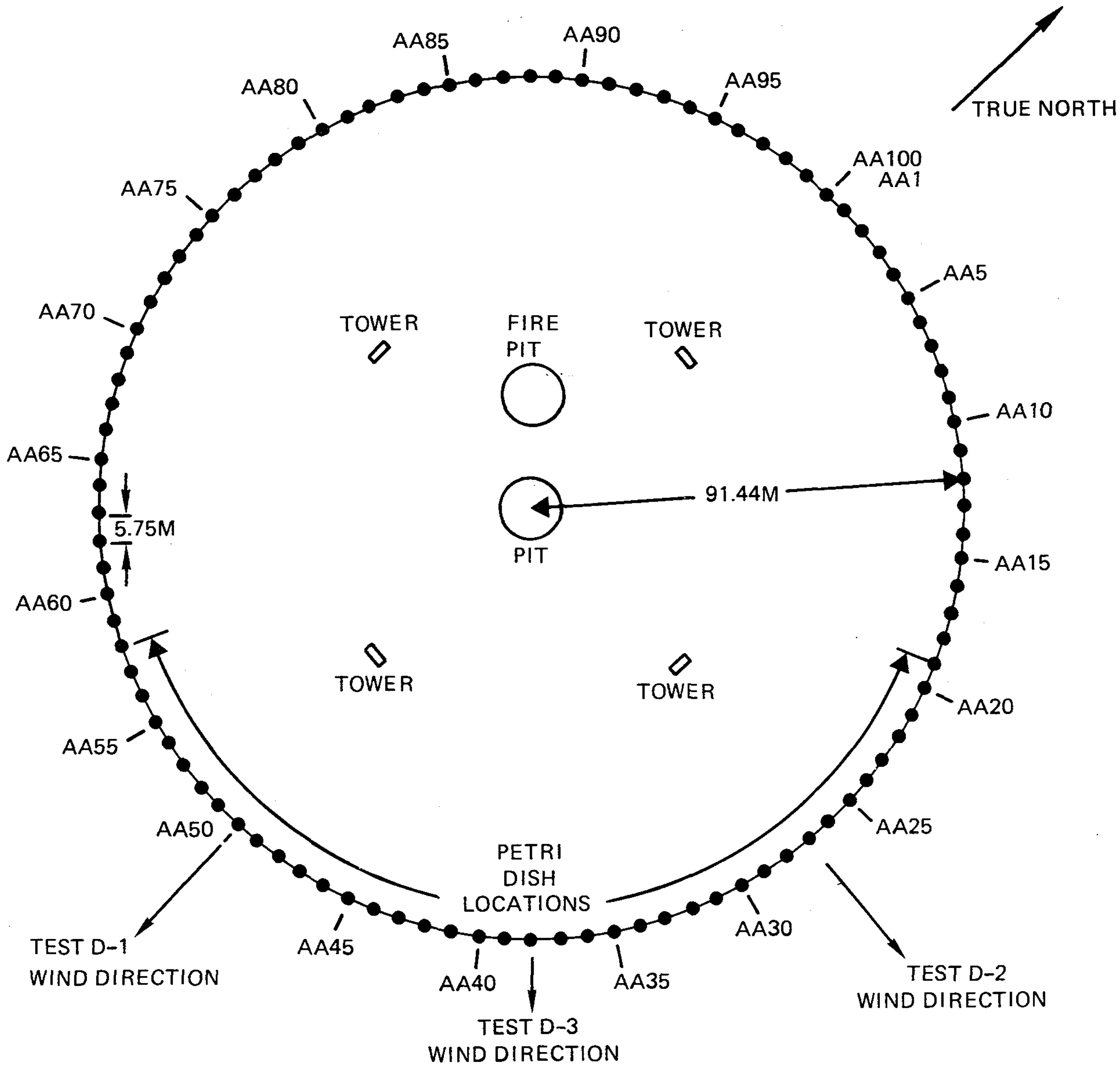


Figure 12. Petri Dish Locations on Dugway Proving Ground Tests

TRW
DEFENSE AND SPACE SYSTEMS GROUP

REPRESENTATIVE FIBRILLATION EFFECTS - I

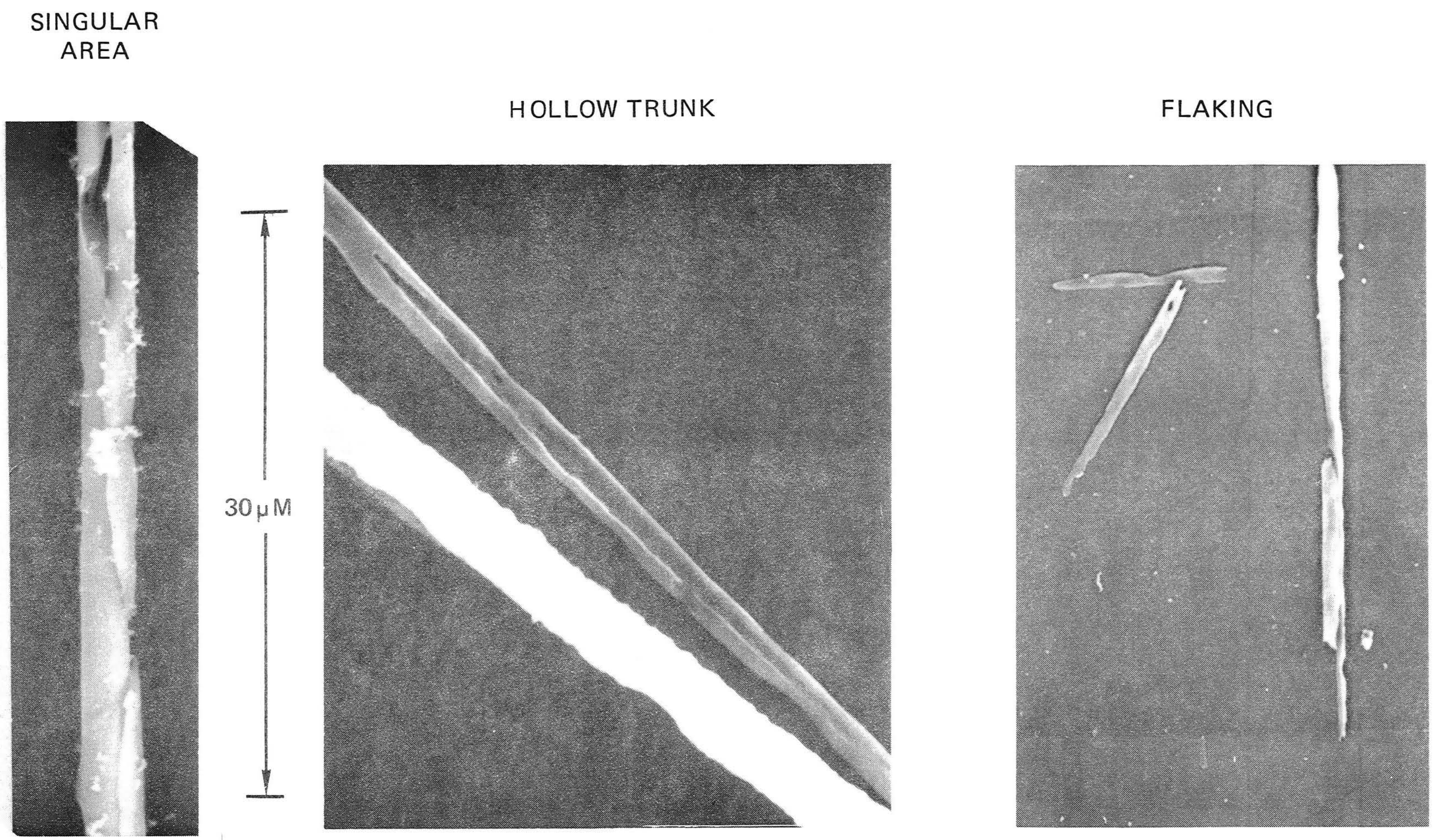


Figure 13. Representative Fibrillation Effects - I

EYE-OF-THE-NEEDLE

SUBMICRON FIBER
(D = 0.4 μ, L = 8.7 μ)

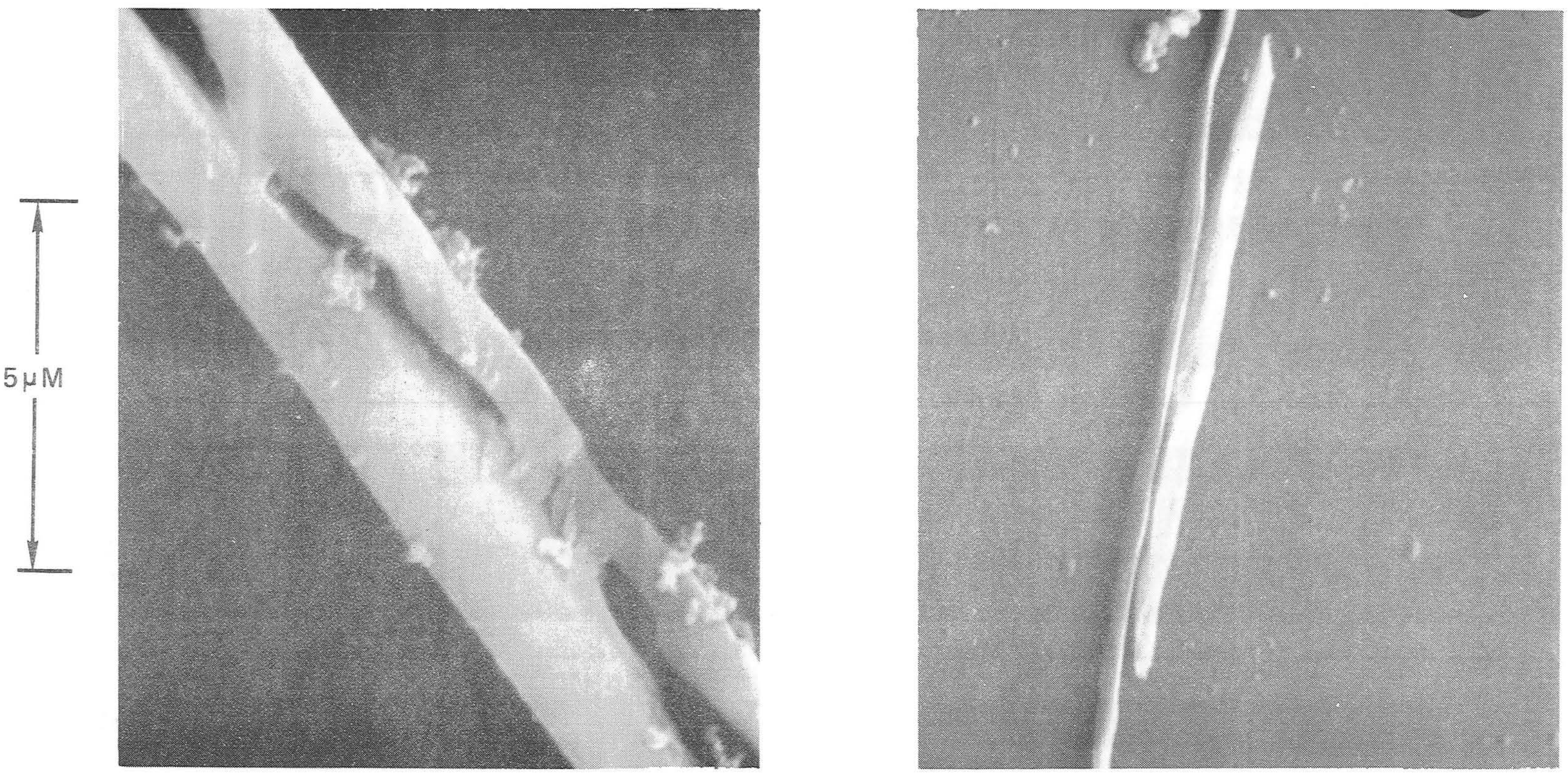


Figure 14. Representative Fibrillation Effects - II

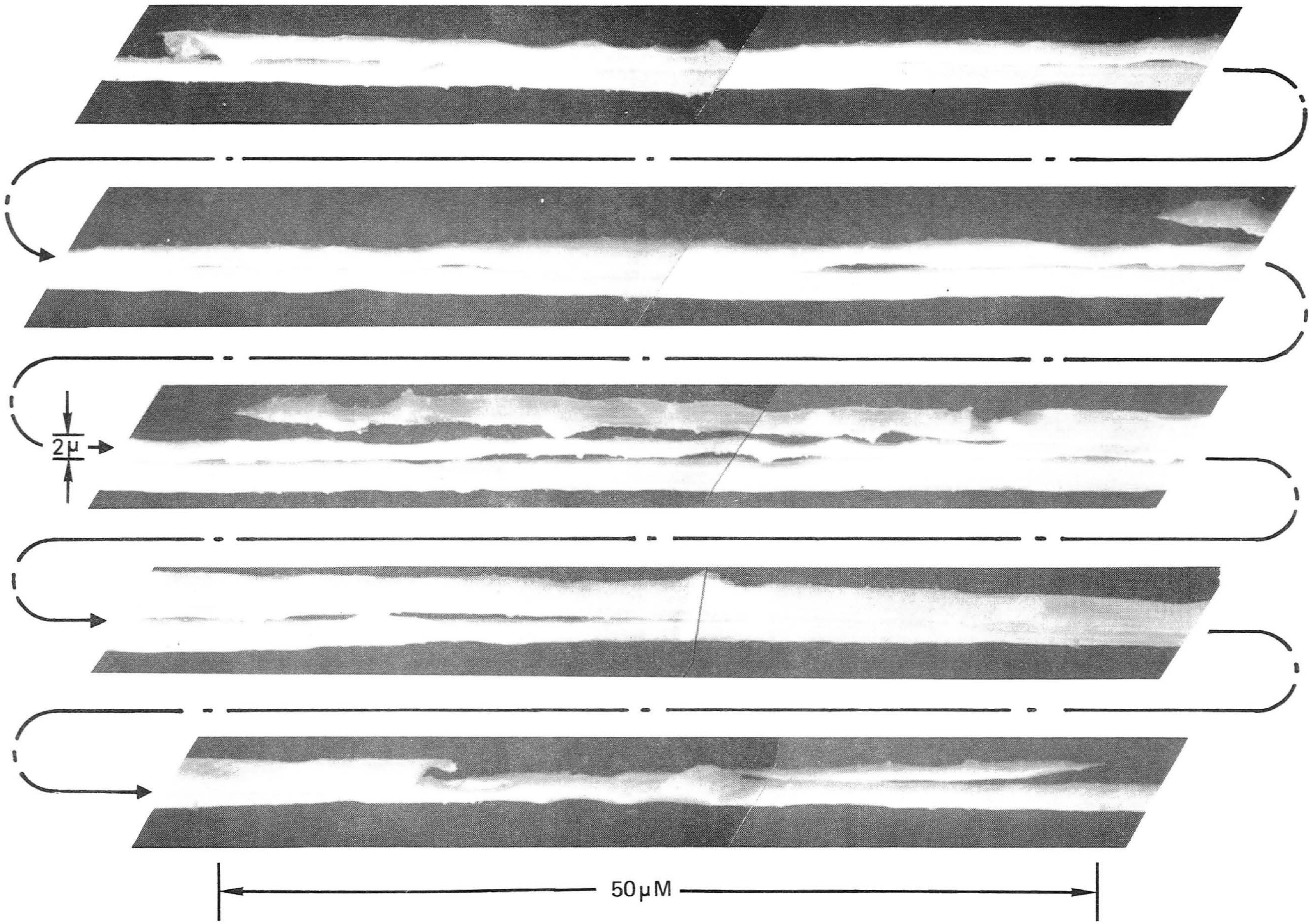


Figure 15. Fibrillation Phenomena Over Extended Length

characteristic of the micron size fibers associated with the fibrillated particles evaluated in the present study.

FIBER MICROSTRUCTURE

The foregoing results indicate that considerable degradation of the fiber structure may be attributed to the high temperature fire environment. There appeared to be singular areas where preferential oxidation occurred with frequent irregular patterns in the fibril evolution. A literature survey [9-15] was performed to gain insight into the original microstructure of the carbon fibers. It appears that significant defects and flaws have been observed in various types of precursors and graphite fibers.

Precursor defects of the following nature have been identified:

- o Small inorganic particles (0.5 - 1 micron)
- o Large organic particles (1-4 microns)
- o Irregular voids due to rapid coagulation
- o Cylindrical voids due to dissolved gases

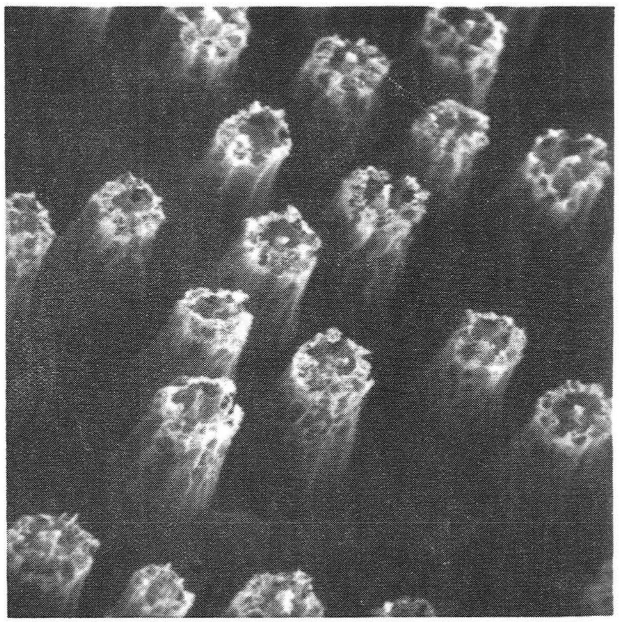
In addition, various types of carbon fiber flaws have been observed such as:

- o Bubble defects due to volatitization of inclusions
- o Mrozowski cracks due to anistrophic thermal contraction during cooldown process
- o Pockets of low crystalline density
- o Large cavities (1-3 microns)

A technique of carbon fiber etching in an oxygen plasma had been developed[9] whereby characteristic patterns of the microstructure may be observed by scanning electron microscopy. A representative SEM photograph for MODMOR I, a high modulus carbon fiber, and an associated three-dimensional structural model [10,11] are shown in Figure 16. A similar microstructure profile has been observed for intermediate modulus carbon fibers but with the irregularities occurring to a lesser extent. The degree and nature of the defects are generally sensitive to the heat treatment temperature of the precursor gaphitization process, with greater manifestation of internal bubbles or voids elongated along the fiber axis for temperatures of the order of 1800^o or greater corresponding to high modulus fibers.

Surface and internal flaws have been observed[12] by means of high-voltage transmission electron microscopy for fibers treated at 1000^{o}C. The internal flaws were attributed to discontinuities in the precursor polymer fiber structure, such as particles or voids with sizes in the range of 0.5 to 3 microns. The concentration per unit length of these flaws were measured for a number of

SEM PHOTOGRAPH OF ETCHED SAMPLE



MICROSTRUCTURE PROFILE

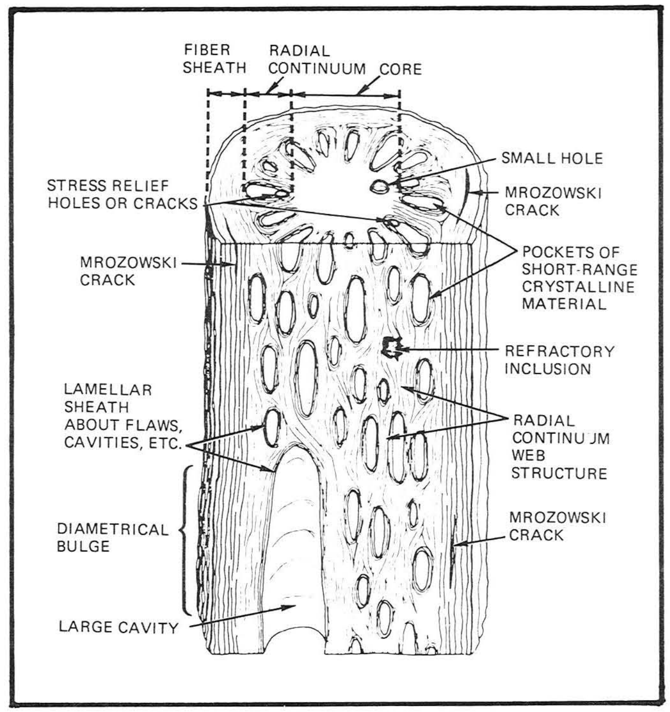


Figure 16. Modmor I Carbon Fiber Structural Model

different samples, with the observed mean flaw concentration varying from 0.03 to 2.3 flaws/mm. It is estimated that there is a sixty percent probability that one serious flaw will occur in each 2.3 cm length of fiber[16]. No direct reference of data of this nature to T-300 carbon fiber was located.

It appears, therefore, that internal flaws in carbon fibers are associated with preexisting and morphologically similar internal flaws in the acrylic precursor (PAN). Structural faults in the textile precursor such as cavities, inorganic particles, organic precipitates, etc. are all incipient flaws and yield corresponding discrete defects in the carbon fiber. In addition, other types of volume flaws may develop as a byproduct of the pyrolysis process.

For the case of pan based carbon fibers of intermediate modulus such as T-300, etch patterns have indicated a radial microstructure with a very thin onion skin structure at the surface. It is quite probable that when enveloped in a fire environment the outer shell is removed during the initial stages of the fiber oxidation process with exposure of the internal radial structure with pockets of low crystalline density and potential voids for subsequent development of fibrillation effects of the type shown in Figures 13 to 15.

FIBER OXIDATION

Physical factors such as volume flaws can affect oxidation resistance. Enhanced oxidation effects can occur in low crystalline density regions. In addition, sodium impurities can cause catalytic oxidation in carbon. Long term testing (hundreds of hours) at 500 to 800^{o}F has indicated that high sodium content is characteristic of poor oxidation resistant fibers. T-300 has been generally categorized as a high sodium content fiber.

As noted earlier arc emission data were obtained of the trace elements contained in spoiler residue and raw T-300 fiber samples for the purpose of a frame of reference toward evaluation of the fibrillated fibers by electron microscopy.

In general, values were quite comparable for all elements except for an anomaly in sodium content, where the spoiler residue was reported with 500 PPM and the T-300 fiber with less than 10 PPM (trace). This result was quite surprising since sources in the literature [19] indicated the sodium content for T-300 to be in the range of 1000 to 2000 PPM.

Additional arc emission data was acquired for other samples containing T-300 fiber with the results shown in Table 21. High sodium content was measured in all cases such that no insight was gained toward the question of the unusually low content for the raw T-300 fiber.

This question was explored with Dr. Roger Bacon and Dr. Myles Towne of union Carbide, Parma, OH. with the result that the answer was readily apparent, namely that there are two types of T-300 fiber, one with high sodium content and the other with low sodium content. The sodium ingredient had been eliminated in the precursor development for the second type. This new material has been distributed by Union Carbide as 6000-filament F-300 yarn for any new purchases during the past 2 years. The original T-300 fiber was produced as a 3000-filament yarn with normal sodium content of about 1500 PPM. This material has been applied to all aerospace and military contracts.

Table 21. T-300 Sodium Content Based on Arc Emission Technique

SOURCE	SAMPLE	SODIUM CONTENT
NWC 11	SPOILER PRE-TEST RESIDUE	500 PPM
AFML	RAW T-300 FIBER	<10 PPM
NWC 1–6	PLATE PRE-TEST RESIDUE	800 PPM
AFML	PLATE RESIDUE	1200 PPM
ITTRI	PLATE RESIDUE	1300 PPM
NARMCO	PRE-PREG	1900 PPM
FIBRITE	PRE-PREG	1700 PPM
AMMRC	UNKNOWN	1000 PPM

All 6000-filament yarn of T-300 on the market is of low sodium content. Examination of the T-300 yarn used for the arc emission data indicated it to be of this type and therefore, resolving the anomaly in the data.

LABORATORY ANALYSIS

It was recognized that the same T-300 (6K) yarn had been used by TRW for TGA tests reported earlier (1) where certain unusual oxidation effects had been observed, and for which at the time no explanation was readily apparent.

A plot is shown in Figure 17 of the TGA mass loss evaluation for various NWC pre-test material samples. The curve corresponding to the raw T-300 fiber is located at the far right of the figure with its lowest portion originating at a temperature which was greater than the tempeartures corresponding to total oxidation of the other composite samples. At the time of the research this result appeared to be quite puzzling since some overlap of the curves would have been expected following complete oxidation of the resin (30% of the composite by weight) at lower temperatures. It was speculated that some form of catalytic oxidation may have been introduced by the resin depolymerization by-products.

A reexamination of the data on the basis of the new information gained regarding the two types of T-300 fiber indicates the simple explanation that the low sodium content 6K-filament yarn is more oxidation resistant and that a direct comparison with the curves for the composites is not applicable.

A sample of raw T-300 (3K) yarn was located and an arc emission test indicated a sodium content of 1800 PPM comparable to the data obtained for the T-300 composite residue samples as shown in Table 21. TGA tests were performed for the T-300 (3K) sample similar to the tests conducted for the data of Figure 17.

The usual procedure for TGA tests is to increase the temperature from ambient to the required peak value at some fixed rate generally of the order of 10°F to 20°F per minute. Time periods of possibly an hour may be involved in reaching temperatures of interest of about 1000°F. This long duration of the heating process was not considered a reasonable simulation of the field test fire environment.

A modification of the TGA procedure had been instituted to afford a better simulation of field test conditions. The TGA equipment consists of a hollow cylindrical oven heated by a series of coils and an independent glass bulb within which the sample is placed in a small platinum boat, with a thermocouple being located inside the boat in close proximity to but not in contact with the sample. A thermostatic cut-off control regulates the oven temperature to the designated settings.

Noramlly the sample is placed in the boat, the mass balance set at 100%, the bulb inserted into the oven and then the heating process initiated. This procedure was modified such that the oven was first heated to the required temperature and then the bulb inserted into the oven chamber. As a result the temperature rose to the required levels in time periods of only about 1 to 1.5 minutes.

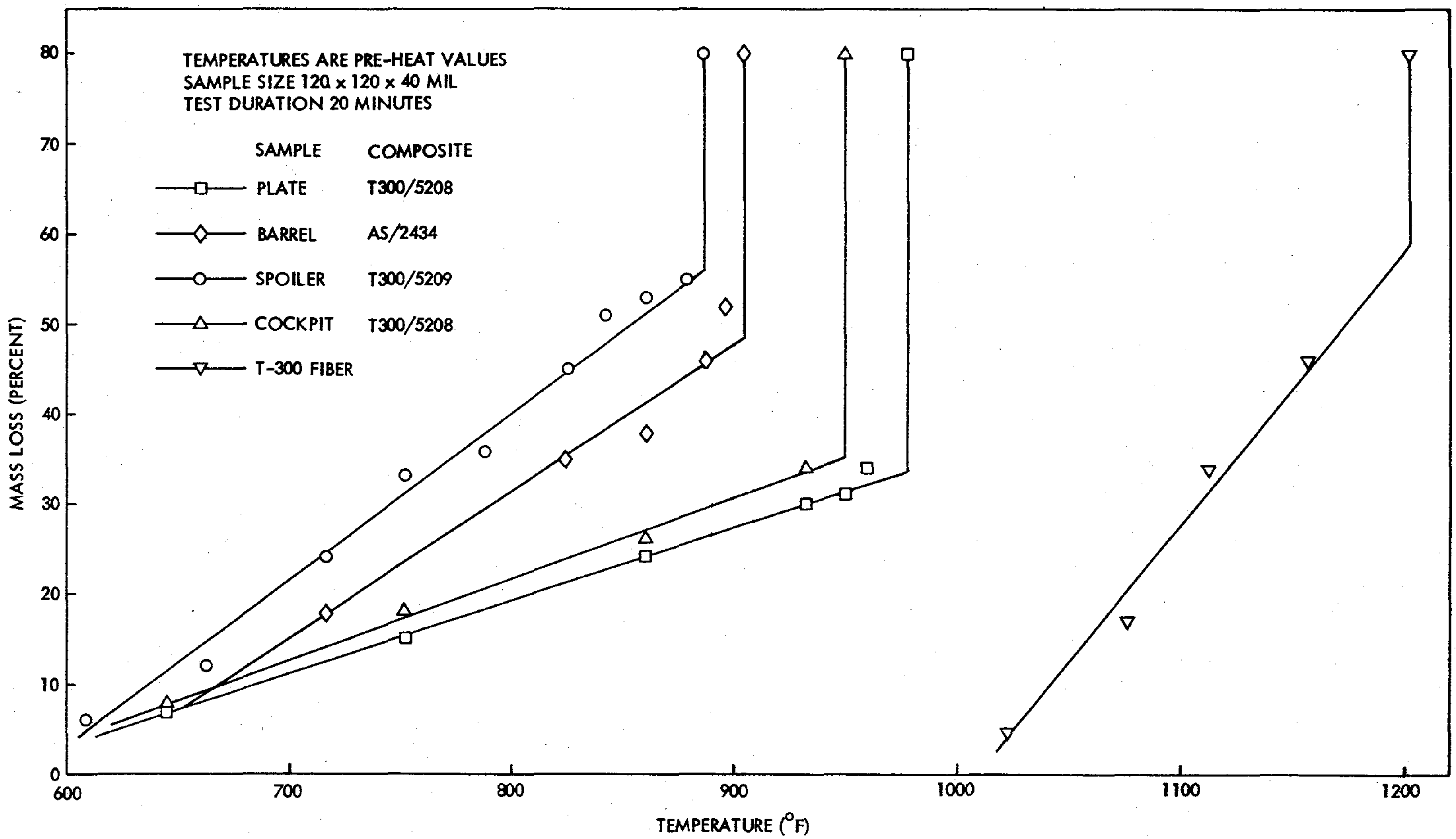


Figure 17. TGA Mass Loss Evaluation For Pre-Test Material Samples

A comparison is presented in Figure 18 of the results of TGA tests of the following materials: (1) NARMCO 5208 and 5209 resin samples, (2) raw T-300 3K and 6K yarns, (3) spoiler and cockpit T-300 residue from resin content acid tests, and (4) NWC plate and cockpit composite (T-300/5208) samples.

There appears to be good agreement among the data for the T-300 3K yarn and for the spoiler and cockpit T-300 residue samples. The single curve designated by T-300 3K yarn and approximating the total set of data is significantly separated toward lower temperatures from the corresponding curve for the T-300 6K yarn, being a manifestation of lower oxidation resistance due to higher sodium content.

The plate and spoiler T-300/5208 composites consisted of 30% resin and 70% carbon fiber by weight. The dashed curve of Figure 18 was developed by multiplication of the ordinate values of the resin curve by 0.3 and summing these products with the products of the ordinate values of the T-300 3K yarn curve by 0.7 at corresponding temperatures. The dashed curve is an estimate of the anticipated mass loss response for the composite samples assuming complete independence in the oxidation of the resin and T-300 fiber. Comparison of the dashed curve with the solid symbols for the plate and spoiler composites indicates excellent agreement, and therefore, eliminates the anomaly of the earlier report as well as the associated conjecture that some degree of catalytic oxidation could be attributed to the resin depolymerization products.

The peak temperatures anticipated in a fire environment are of the order of 1600°F to 2000°F as indicated by the thermocouple data for NWC 11 shown in Figure 19. Estimates of the oxidation time for a single T-300 fiber of the 6K-filament type are plotted in Figure 20, with values ranging from 2.9 minutes for 1600°F to about 20 seconds for 2000°F. Although specific calculations of this nature have not been performed for the 3K-filament type it is estimated that the exposure time for complete oxidation of a single fiber would be lower by a factor of the order of 3 to 5. Therefore, substantial oxidation with associated fibrillation effects can occur over very short time periods in a fire environment.

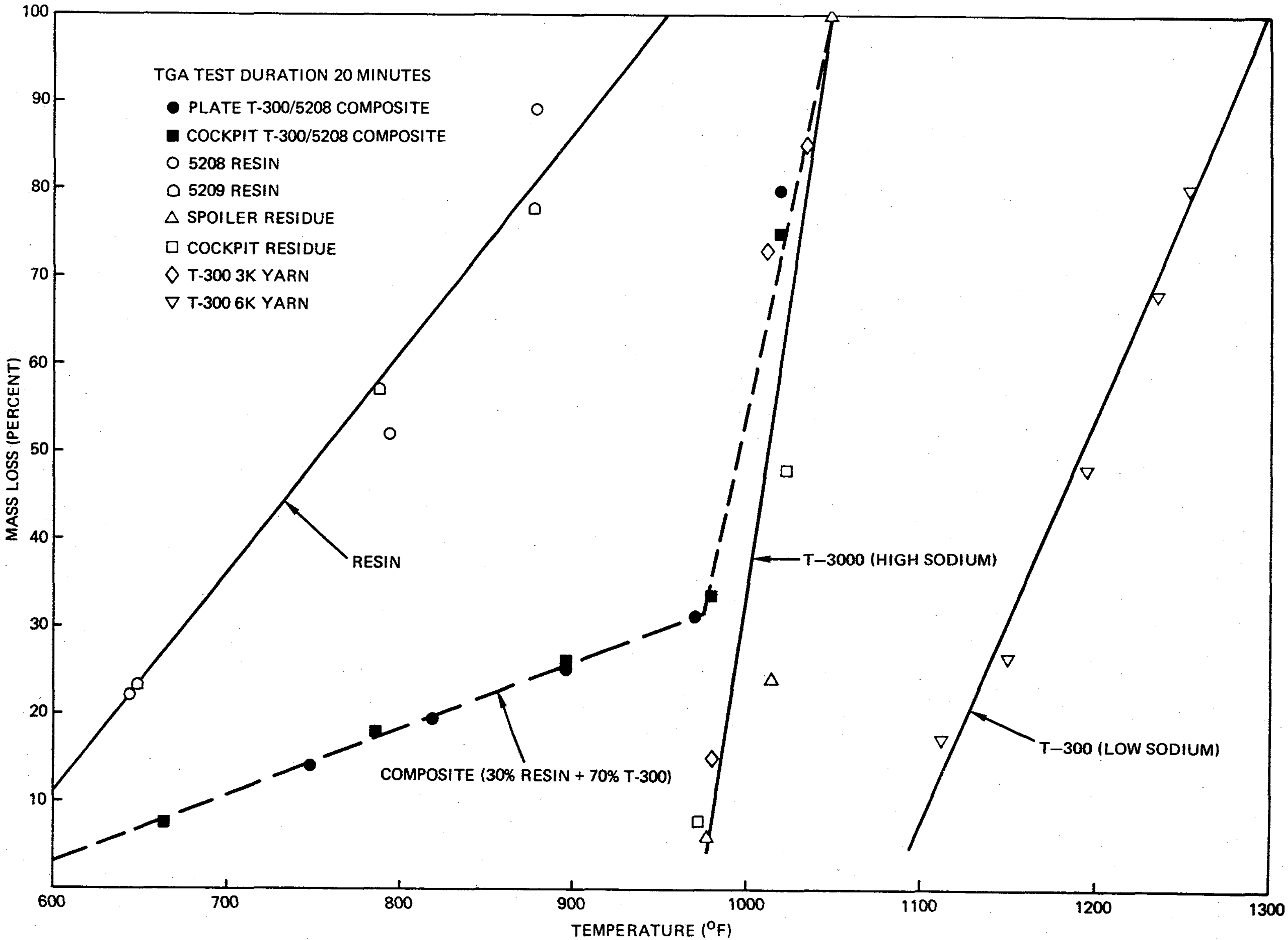


Figure 18. TGA Mass Loss Evaluation

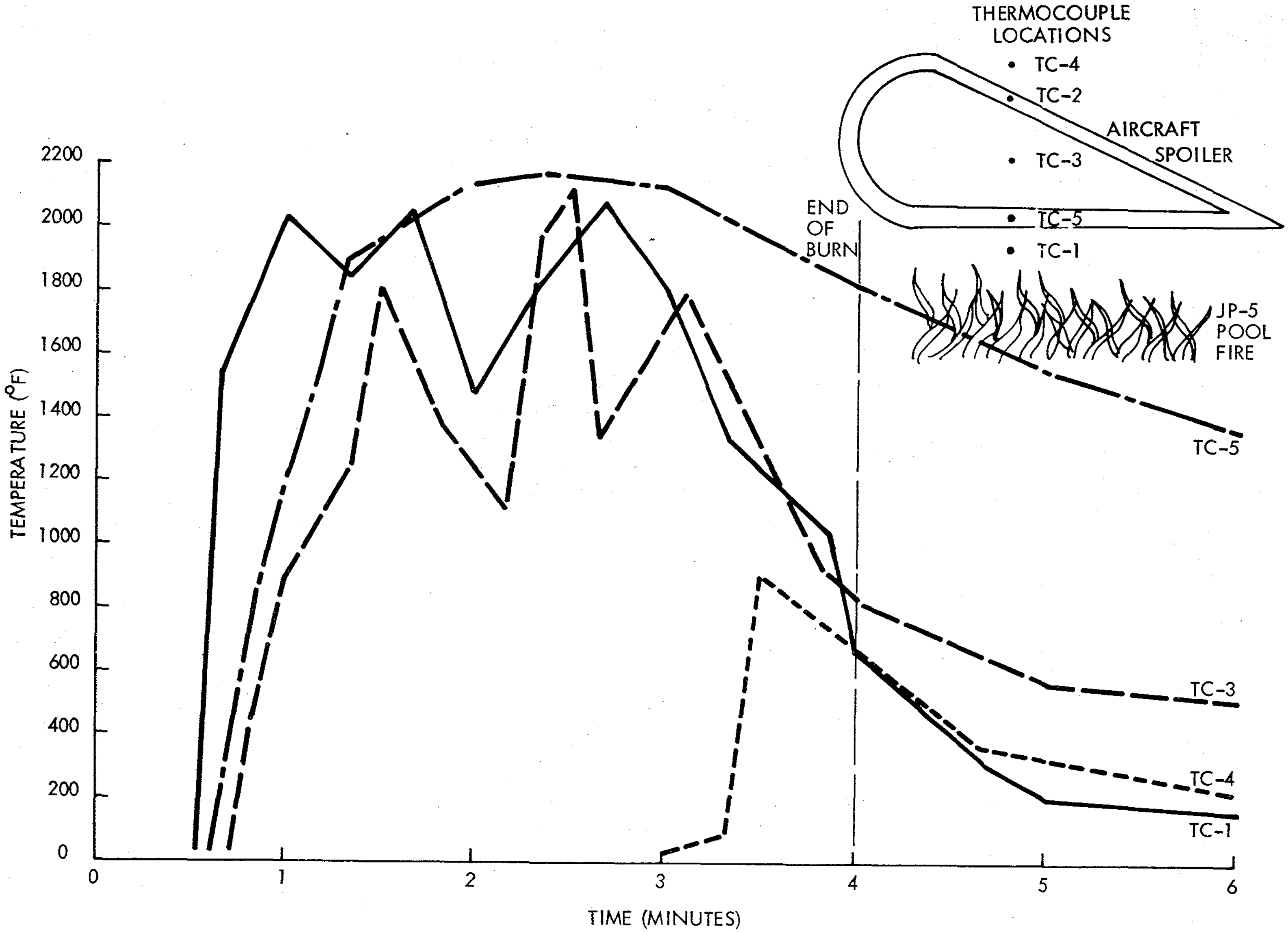


Figure 19. Temperature History During Spoiler Burn, Test No. 11

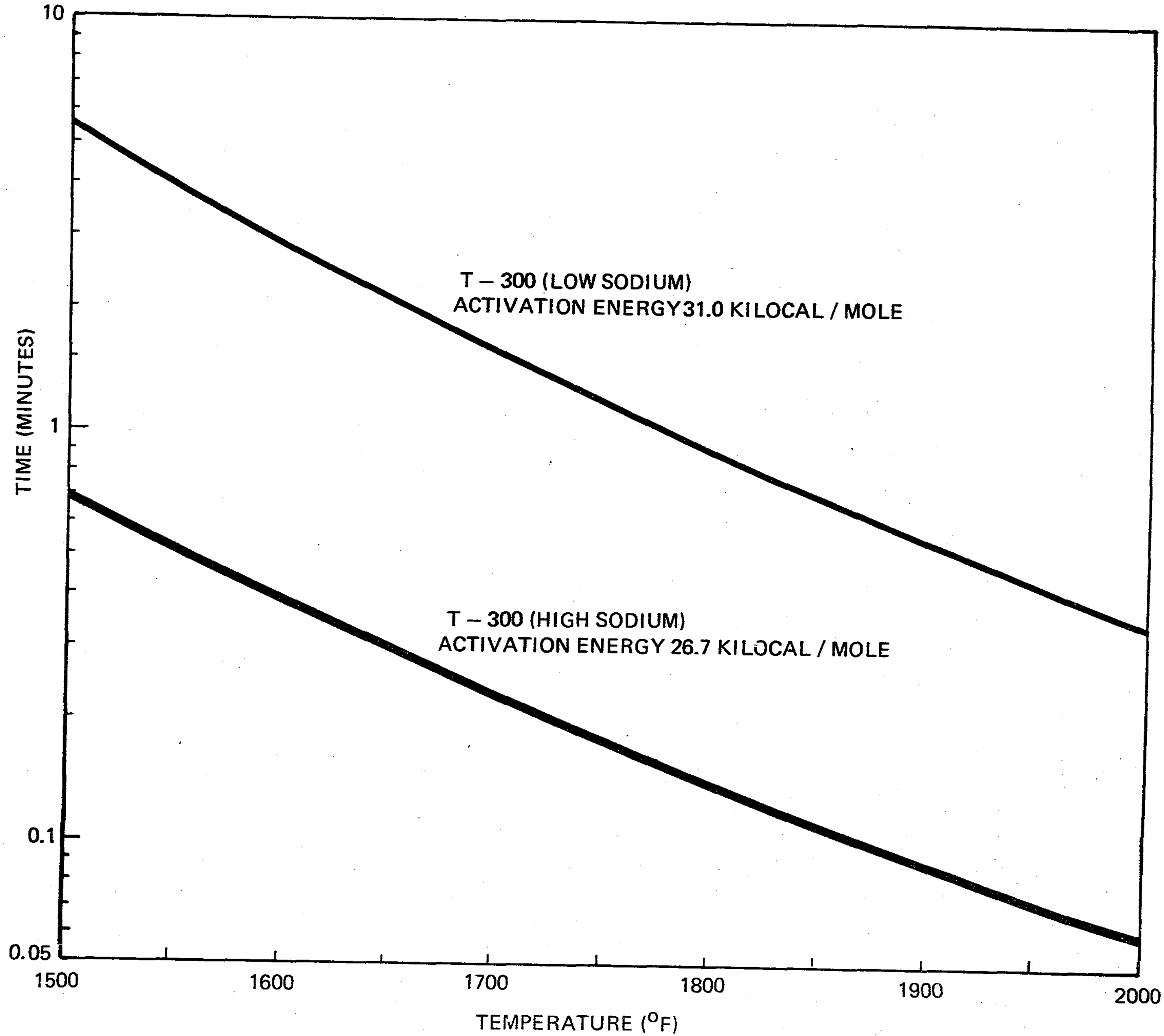


Figure 20. Oxidation Time for Single T-300 Fibers

9. CRITERIA ESTIMATES

Development of micron fiber criteria estimates was based on the following methodology:

1. Assume the NASA-recommended criteria for single fibers with lengths greater than or equal to 1 mm ($N_{L \geq 1mm}$)
2. Determine the frequency ratio of micron fibers to single fibers with lengths greater than or equal to 1 mm.
   - Establish an empirical frequency ratio (FR) from NWC 11 and NWC 13 test data
   - Estimate an enhancement factor (EF) to account for data base limitations
3. Multiply the respective factors to yield the micron fiber criteria as follows:

$$N_{MF} = (N_{L \geq 1mm}) \times (FR) \times (EF)$$

Figure 21 summarizes the NASA recommended criteria[3] on accidental carbon fiber release to be applied for risk analysis computations. Of the total amount of carbon fiber in composite parts exposed to fire 1 percent will be released in the case of accidents with fires and no explosions. For every kilogram of carbon fiber released there will be 5×10^9 single fibers with lengths greater than or equal to 1 mm.

As noted earlier and indicated in Table 18, the frequency ration of micron fibers to single fibers of lengths greater than or equal to 1 mm is estimated to be a factor of 10. However, there are various limitations and uncertainties regarding the data base available for the purpose of establishing this ratio.

A number of these limitations are briefly outlined as follows:

- Considerable difficulty was encountered in the data reduction of isolated micron fibers due to the sparcity of occurrence to the extent that data of this nature was essentially discounted.
- Analytical results encompass only data from close-in ranges with no basis for judgement regarding downrange characteristics.
- Fibrillated fibers constituted the principal source of micron fiber data with the relative frequency of occurrence of high density micron fiber regions as somewhat indefinite.
- Micron fiber criteria have been evaluated only on the basis of NWC test data.

- COMMERCIAL AIRCRAFT ACCIDENT RECORDS INDICATE:
  - 85 PERCENT OF ACCIDENTS WITH FIRES HAVE NO EXPLOSIONS
  - 15 PERCENT OF ACCIDENTS WITH FIRES DO HAVE EXPLOSIONS

- OF TOTAL AMOUNT OF CF IN COMPOSITE PARTS EXPOSED TO FIRE:
  - 1 PERCENT WILL BE RELEASED FROM FIRES ALONE
  - 3-1/2 PERCENT WILL BE RELEASED FROM FIRES AND EXPLOSIONS

- FOR EVERY KILOGRAM OF CF RELEASED:
  - 5×10^9 SINGLE FIBERS WILL BE RELEASED
  - EXPONENTIAL DISTRIBUTION OF FIBER LENGTH WITH MEAN OF 2 MILLIMETERS
  - FIBER DIAMETERS SAME AS ORIGINAL ($8\,\mu$M)

Figure 21. NASA Recommended Accidental Carbon Fiber Release For Risk Analysis Computations

- Prediction of the extent of fibrillation effects associated with aircraft accident scenarios is highly uncertain.

- Potential impact of significant diameter reductions at the micron fiber ends may warrant consideration.

- Data collection technique involving pressure of protective acetate cover sheet over sticky paper adhesive surface may have contributed toward separation of micron fibers from parent fibrillated particles.

- Appropriate scaling factors for increased composite mass and complex structural configurations are unavailable.

- Micron fiber source enhancement due to wind conditions, explosions, turbulence and mechanical disturbances has not been evaluated.

On the basis of the foregoing limitations it appears reasonable to introduce an enhancement factor of 10 in the frequency ratio of micron fibers to single fibers with lengths greater than or equal to 1 mm.

Micron fiber criteria estimates based on the analyses of the present study are presented in Figure 22. The total number of micron fibers per kilogram mass of carbon fiber released is specified as 5×10^{11}, with a corresponding micron fiber mass fraction of carbon fiber released as 5%. The spectral distribution of micron fiber dimensions reflects a relatively uniform pattern over the respective diameter and length intervals.

The criteria values of Figure 22 constitute a description of the micron fibers generated at the source during an aircraft accident involving a fire only. It appeared of interest to evaluate an extreme case estimate of the potential exposure to the micron fiber plume propagating downwind. Results of the upper limit estimates are shown in Figure 23.

The mass of carbon fiber exposed to the fire was assumed as 1000 Kg resulting in a total number of 5×10^{12} micron fibers released. A schematic of the carbon fiber plume profile is shown in Figure 23. For the purpose of the present analysis no consideration is given to the particle rainout occurring in the close-in regions, with attention focussed principally on exposures beyond the plume cross-section designated in Figure 23 as downrange source area. No loss of micron fibers is considered for the close-in areas with the total source of 5×10^{12} micron fibers assumed available for propagation downrange. In addition, reduction of concentration levels due to dispersion of the fiber cloud is neglected.

The peak concentration level evaluated at the position of the downrange source area is given by the relation:

$$C = \frac{N_f}{\frac{\pi}{4} D_s^2 V_w t_b}$$

- MICRON FIBER FREQUENCY RATIO RELATIVE TO SINGLE FIBER LENGTHS $\geq$ 1MM - - - - 10
- ENHANCEMENT FACTOR TO ACCOUNT FOR LIMITATIONS AND UNCERTAINTIES - - - - 10
- MICRON FIBERS PER KILOGRAM MASS OF CARBON FIBER RELEASED
  - ASSUME NASA VALUE FOR LENGTHS $\geq$ 1MM = 5×10^9 FIBERS
  - MULTIPLY BY MICRON FIBER FREQUENCY RATIO AND ENHANCEMENT FACTOR

    NUMBER OF MICRON FIBERS = $\left(5 \times 10^9\right) \times \left(10\right) \times \left(10\right) = 5 \times 10^{11}$
- MICRON FIBER DIMENSIONS

LENGTH (MICRONS)	PERCENT OF TOTAL	DIAMETER (MICRONS)	PERCENT OF TOTAL
2 – 10	10	0.4 – 1.0	21
10 – 20	24	1.0 – 1.5	27
20 – 30	21	1.5 – 2.0	20
30 – 40	16	2.0 – 2.5	23
40 – 60	16	2.5 – 3.0	9
60 – 80	13	–	–

- MICRON FIBER MASS PER KILOGRAM OF CARBON FIBER RELEASED
  - AVERAGE MASS PER FIBER: $\overline{M} = \frac{\pi}{4}\,\overline{D}^2\,\overline{L}\,\rho = \frac{\pi}{4}\left(1.5 \times 10^{-4}\right)^2 \left(30 \times 10^{-4}\right)\left(1.8\right) = 1 \times 10^{-10}\ G$
  - TOTAL MICRON FIBER MASS: $M_T = \left(5 \times 10^{11}\right)\left(1 \times 10^{-10}\right) = 50\ G$
  - MASS FRACTION OF CF RELEASED: 5 PERCENT

Figure 22. Micron Fiber Criteria Estimates

- MASS OF CF EXPOSED TO FIRE — 1000 KG
- MASS OF CF RELEASED (1%) — 10 KG
- NUMBER OF MICRON FIBERS RELEASED — 5×10^{11} PER KG
- TOTAL MICRON FIBERS RELEASED — 5×10^{12}
- CONCENTRATION $C = \dfrac{N_f}{\frac{\pi}{4} D_s^2 V_w t_b}$

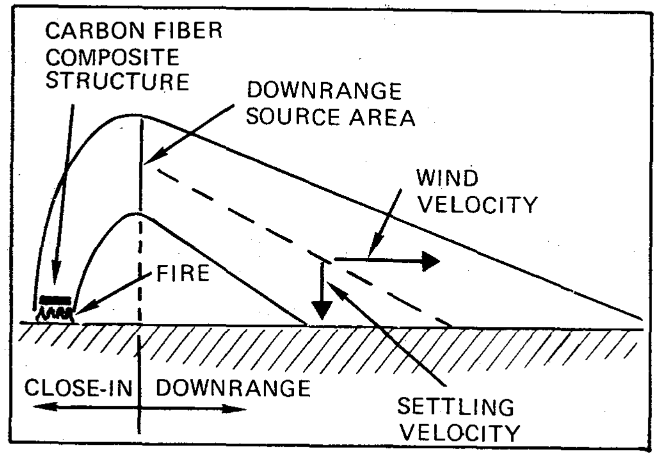
  N_f = FIBER NUMBER

  $\frac{\pi}{4} D_s^2$ = SOURCE AREA

  V_w = WIND VELOCITY

  t_b = BURN TIME

CARBON FIBER PLUME PROFILE



- PARAMETER ASSUMPTIONS

  $D_s = 200\text{M} \quad V_w = 0.5\ \text{M/SEC} \quad t_b = 60\ \text{SEC}$

  $$\text{CONCENTRATION} = \frac{5 \times 10^{12}}{\frac{\pi}{4}(200)^2\,(0.5)(60)} = 5.3 \times 10^6\ \text{F/M}^3$$

  EXPOSURE = CONCENTRATION X BURN TIME

  $$= (5.3 \times 10^6)(60) = 3.2 \times 10^8\ \frac{\text{F} - \text{S}}{\text{M}^3}$$

- OSHA ASBESTOS STANDARD: CONCENTRATION $2 \times 10^6\ \dfrac{\text{F}}{\text{M}^3}$

  EXPOSURE $5.8 \times 10^{10}\ \dfrac{\text{F–S}}{\text{M}^3}$ PER 8-HR DAY (CUMULATIVE)

Figure 23. Upper Limit Estimate of Micron Fiber Exposure

where N_f = total micron carbon fiber number

$\frac{\pi}{4} D_s^2$ = plume cross-sectional area

V_w = wind velocity

t_b = burn duration

The plume diameter D_s at the source area was estimated to be 200 meters. In order to maximize the concentration level the wind velocity is assumed as 1 mph (0.5 m/sec) and burn duration as 1 minute.

The peak carbon fiber concentration level is therefore:

$$(C_{CF})_{max} = \frac{5 \text{ X } 10^{12}}{\frac{\pi}{4}(200)^2(0.5)(60)} = 5.3 \text{ X } 10^6 \text{ f/m}^3$$

The maximum carbon fiber exposure determined by $E = C\ t_b$ is therefore:

$$(E_{CF})_{max} = \left(5.3 \text{ X } 10^6\right)(60) \doteq 3.2 \text{ X } 10^8 \text{ f-s/m}^3$$

The corresponding values for the OSHA asbestos standard are:

$$(C_{AS})_{max} = 10^7 \text{ f-s/m}^3 \text{ (ceiling concentration)}$$

$$E_{AS} = 5.8 \text{ X } 10^{10} \text{ f-s/m}^3 \text{ per 8-hr. day (cumulative)}$$

The OSHA asbestos exposure level is based on a permissible concentration level of $2 \text{ X } 10^6$ f/m^3 as a time-weighted average over a single 8-hour period ($2.9 \text{ X } 10^4$ sec.). The exposure level is cumulative since OSHA criteria permit similar exposures for each successive work day.

The upper limit of the micron carbon fiber concentration level is only about half the permissible OSHA asbestos ceiling concentration level. Consideration of factors such as higher wind velocities, longer burn times and downrange plume dispersion would lead to values even lower than the OSHA asbestos standard. The estimated peak micron carbon fiber exposure level of $3.2 \text{ X } 10^8$ fiber - seconds per cubic meter corresponds to an exposure time of 160 seconds at the OSHA asbestos concentration level of $2 \text{ X } 10^6$ f/m^3. It appears reasonable to conclude that the micron fiber exposure levels resulting from an aircraft accident would be substantially lower than the OSHA standard criteria for asbestos.

The above estimates were based on consideration of aircraft fires only. According to the NASA recommended criteria of Figure 21 an increase in the values by a factor of 3.5 would be required for values corresponding to aircraft accidents with a fire and explosion.

10. CONCLUSIONS

Principal highlights of the study are briefly summarized as follows:

- Fibrillation Phenomena

  - Fibrillated fibers were observed during the course of data reduction of NWC sticky paper records with a relative frequency of occurrence of about 20%.

  - This type of fiber constituted the principal source of micron carbon fibers of interest toward development of health hazard criteria.

  - Conclusive evidence of the fibrillation phenomena being attributed to single carbon fibers was obtained as a result of evaluation of Petri dish data from DPG tests by means of SEM microscopy.

  - Potential carbon fiber surface and volume defects may contribute to the evolution of fibrillation effects in addition to catalytic oxidation by sodium impurities.

- Micron Carbon Fiber Characteristics

  - Relative occurrence of micron carbon fibers ($D < 3$ μm, $L < 80$ μm) is estimated to be 60%, with a frequency ratio of 10 relative to single fibers with lengths greater than or equal to 1 mm.

  - Data base limitations and uncertainties warrant introduction of an enhancement factor of 10 increasing the estimated frequency ratio to 100.

  - The weighted average of the micron carbon fiber distribution indicated an average diameter of 1.5 microns and an average length of 30 microns.

- Micron Fiber Criteria

  - The number of micron carbon fibers generated per kilogram of carbon fiber released during an aircraft accident is estimated to be 5×10^{11}, with a mass fraction of 5% of the total fiber released.

  - An extreme case analysis for an aircraft fire has yielded an upper bound in micron carbon fiber concentration level of approximately 5×10^6 f/m^3 as compared to the OSHA asbestos concentration of 10^7 f/m^3.

- An upper limit exposure to carbon fibers is estimated to be about 3.2×10^8 f-s/m^3 which corresponds to exposure to the OSHA 8-hr. day asbestos concentration level for a period of about 160 seconds.

- Micron carbon fiber exposure levels are anticipated to be substantially lower than the cumulative OSHA asbestos value of 5.8×10^{10} f-s/m^3 per 8-hr. day.

11. REFERENCES

1. Lieberman, P; Chovit, A. R.; Sussholz, B.; and Korman, H. F.: Data Reduction and Analysis of Graphite Fiber Release Experiments. (TRW Defense and Space Systems Group; NASA Contract NAS 1-15465.) NASA CR-159032, 1979.

2. Pride, R. A.: Large-Scale Fiber Release and Equipment Exposure Experiments. NASA Conference Publication 2119, 1980, pp. 101-136.

3. Bell, Vernon L.: Release of Carbon Fibers From Burning Composites. NASA Conference Publication 2119, 1980, pp. 29-57.

4. Occupational Safety and Health Administration: General Industry Safety and Health Standards. OSHA 2206, Code of Federal Regulations, Title 29, Part 1910.93a, July 1972.

5. Occupational Safety and Health Administration: General Industry Safety and Health Standards. OSHA 2206, Code of Federal Regulations, Title 29, Part 1910.1001, Revised January 1976.

6. Occupational Safety and Health Administration: Occupational Exposure to Asbestos. Federal Register, Part II, 9 October 1975.

7. National Institute for Occupational Safety and Health: Revised Recommended Asbestos Standard. DHEW (NIOSH) Publication No. 77-169, December 1976.

8. National Institute for Occupational Safety and Health: Criteria for a Recommended Standard Occupational Exposure to Fibrous Glass. DHEW (NIOSH) Publication No. 77-152, April 1977.

9. American Conference of Governmental Industrial Hygienists: Threshold Limit Values for Chemical Substances and Physical Agents in the Workroom Environment with Intended Changes for 1978. 1978.

10. American Conference of Governmental Industrial Hygienists: Documentation of the Threshold Limit Values for Substances in Workroom Air. 1978.

11. British Advisory Committee on Asbestos of the Health and Safety Commission and Ministers: Volume 1: Final Report of the Advisory Committee, and Volume 2: Papers Prepared for the Advisory Committee. 1979.

12. Barnet, F. Robert; and Norr, Marriner K.: Carbon Fiber Etching in an Oxygen Plasma . Carbon, vol. 11, 1973, pp. 281-288.

13. Barnet, F. Robert; and Norr, Marriner K.: A Three-Dimensional Structural Model for a High Modulus Pan - Based Carbon Fiber. Naval Ordnance Laboratory NOLTR 73-154, June 1974.

14. Barnet, F. Robert; and Norr, Marriner K.: Factors Limiting the Tensile Strength of Pan - Based Carbon Fibers. Naval Ordnance Laboratory NOLTR 74-172, November 1974.

15. Sharp, J. V.; and Burnay, S. G.: High-Voltage Electron Microscopy of Internal Defects in Carbon Fibers. International Carbon Fibers Conference, London, 2-4 February 1971, Paper 10, pp. 68 - 72.

16. Johnson, J. W.; and Thorne D. J.: Effects of Internal Polymer Flaws on Strength of Carbon Fibers Prepared from Acrylic Precursor. Carbon, vol. 7, 1969, pp. 659 - 661.

17. Thorne, D. J.: Distribution of Internal Flaws in Acrylic Fibers. Journal of Applied Polymer Science, vol. 14, 1980, pp. 103 - 113.

18. Jones, W. R.; and Johnson, J. W.: Intrinsic Strength and Non-Hookean Behavior of Carbon Fibers. Carbon, vol. 9, 1971, pp. 645-655.

19. McMahon, P.E.: Advanced Composite Materials - Environmental Effects. ASTM Special Technical Publication No. 658, September 1977, pp. 254 - 266.

1. Report No. NASA CR-159217	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle Evaluation of Micron Size Carbon Fibers Released from Burning Graphite Composites		5. Report Date April 1980
		6. Performing Organization Code
7. Author(s) B. Sussholz		8. Performing Organization Report No. NASA/TRW 80-06
9. Performing Organization Name and Address TRW DSSG 1 Space Park Redondo Beach, CA. 90278		10. Work Unit No.
		11. Contract or Grant No. NAS 1-15465
12. Sponsoring Agency Name and Address NASA Langley Research Center Hampton VA. 23665		13. Type of Report and Period Covered Contractor Report
		14. Sponsoring Agency Code
15. Supplementary Notes		

16. Abstract Quantitative estimates were developed of micron carbon fibers released during the burning of graphite composites. Evidence was found of fibrillated particles which were the predominant source of the micron fiber data obtained from large pool fire tests. The fibrillation phenomena was attributed to fiber oxidation effects caused by the fire environment. Analysis of propane burn test records indicated that wind sources can cause considerable carbon fiber oxidation. Criteria estimates were determined for the number of micron carbon fibers released during an aircraft accident. An extreme case analysis indicated the upper limit of the micron carbon fiber concentration level was only about half the permissible OSHA asbestos ceiling concentration level.

17. Key Words (Suggested by Author(s)) Carbon Fibers Micron Size Particles Health Hazard		18. Distribution Statement Unclassified - Unlimited	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 81	22. Price*

For sale by the National Technical Information Service, Springfield, Virginia 22161

DO NOT REMOVE SLIP FROM MATERIAL

Delete your name from this slip when returning material to the library.

NAME	MS
~~Navy Environmental Health Center~~	~~IL~~
~~3 week loan~~	