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Project Summary

Monitoring Techniques for Carbon Fiber Emissions: Evaluation B

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Carbon fibers released from manufacturing, application and waste disposal operations are light in weight and can be dispersed over wide areas. Because of their high electrical conductivity, they can damage any electronic apparatus and electrical equipment they contact. The impact of respirable fibers on health is not known. The EPA has the responsibility to evaluate and develop instrumentation for continuously monitoring the number and mass of carbon fibers emitted from source operations. The current program was conducted to evaluate available measurement methods in light of source emission characteristics.

Carbon fibers released during manufacturing and application are generally well controlled by exhaust hoods and filters. Major emission points include tow rewind, chopping, textile weaving and machining operations. The range of fiber concentration and length distribution is large; other particulate matter, including other types of fibers, is frequently present.

A total of 11 candidate monitoring methods based on contact (electrical), locally sensing (optical, microwave) and remote sensing (microwave, radar) were identified. Each method was rated on the basis of measurement (sampling), detection and instrumental parameters, and their fit with fiber concentration and length ranges produced by three emission scenarios representing textile weaving, machining and waste incineration. Five methods have merit for certain conditions and are recommended for further study: for moderate to high concentrations and lengths > 1 mm, microwave-OSGEF and electrical grid-arc methods; for moderate to high concentrations and lengths <1 mm, optical scattering-rotating lens and fiber aerosol monitor (FAM) methods; and for very high concentrations in absence of other particulate matter (i.e., process upset), the optical-LED method. Microwave-OSGEF is the only method that is specific to carbon fibers. The electric grid-arc method can be arranged to sample a major portion of the air stream, providing representative sampling. These two methods are recommended as having highest priority for further development.

This Project Summary was developed by EPA's Environmental Sciences Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Carbon fiber-epoxy composites are extremely stiff and strong relative to their weight. They have been used during the past decade as a structural reinforcement material by the aerospace industry and, more recently, for recreational equipment. A 25 to 40 percent annual increase in use is expected over the next decade, and, depending on their future use in automobiles, the growth rate could be considerably more. Because of their high electrical conductivity, carbon fibers released into the atmosphere can damage nearby electronic and electrical equipment. The fibers are small and lightweight and can disperse over fairly long distances.

The relatively few manufacturers of carbon fibers and composite materials are aware of this hazard; they use in-plant pollution control procedures and dispose of waste materials in landfill. Nevertheless, there have been accidental releases to the atmosphere.

EPA has the responsibility to evaluate and develop instrumentation for continuously monitoring the number and mass of carbon fibers emitted from operations such as the manufacture, processing and disposal of carbon fibers or fiber containing materials. The first phase of work which is reported here relates to an evaluation of currently available instruments for monitoring carbon fiber emissions. In this context, carbon fibers are defined as having at least a 5 to 1 length to diameter ratio. Manufactured carbon fibers are taken to be $\geq 5\ \mu m$ in diameter, whereas incompletely incinerated carbon fibers can have diameters $<5\ \mu m$. The program requirements are as follows:

- The evaluation of instruments must determine their applicability to stacks or ducting to stacks (i.e., confined emissions) and their ability to continuously measure mass and number of carbon fibers in the confined emissions from the manufacture, processing, and disposal of such fibers or fiber-containing material.
- Evaluation of performance capability shall address the following operating parameters: accuracy, range, reproducibility, response time, sensitivity, specificity, stability. Test procedures to examine these parameters shall be presented.
- A work plan shall be prepared and delivered to the EPA describing the recommended monitoring system. The work plan shall include whatever modifications and new developments are needed to provide an optimized prototype system for field evaluation.

The approach to establishing the required information base was as follows:

- Develop a data base on carbon fiber manufacturing and application with consideration to
  - Unit processes
  - Carbon fiber emission points
  - Control methods
  - Available sampling data.
- From this data base, describe the characteristics of typical emission scenarios for carbon fiber manufacturing, use and disposal.
- Identify instrumental methods potentially useful for monitoring carbon fiber emissions on the basis of literature review and interviews with government agencies and contractors.
- Rank candidate monitor methods on the basis of performance criteria according to the selected emission scenarios.
- Based on the above, recommend modifications and new development efforts required for a monitoring method to meet the EPA needs.

Initially, the effort was directed toward evaluating presently available instrumentation; however, based upon certain shortcomings of all candidate monitor instruments, an additional task was added to the program to carry out a laboratory evaluation of an Arthur D. Little, Inc., monitoring concept. The new concept is based on an optical signal resulting from interaction of carbon fibers with a high frequency electric field, referred to herein as the OSGEF method.

Manufacture of Carbon Fiber Composites

Process Description

Carbon fibers are made from precursors such as resins, hydrocarbon pitches, lignin pitches, rayon, acrylic polymers, etc. Regardless of the precursor used, processing carbon fibers involves a series of heat treating steps to temperatures which for some fibers may reach 3000OC. A process flow sheet is given in Figure 1.

Carbon Fiber Emission Scenarios

To permit a ranking of monitoring devices and concepts on the basis of their ability to detect and quantify carbon fibers, it is necessary to estimate the characteristics of emission streams containing typical carbon fiber. On the basis of the review undertaken in this contract and previous studies of the emissions from municipal incinerators, three typical carbon fiber emission scenarios have been developed. They are summarized in Table 1.

For scenario A, a carbon fiber textile weaving process conducted in an isolated room equipped with a HEPA filter air cleaning system, the monitoring point could be anywhere between the emission point and the HEPA filter.

In scenario B, a carbon fiber composite machining process, such as grinding, much higher carbon fiber concentrations may be present. Other particulates may also be present at a level about equal to the number of carbon fibers.

Finally, in scenario C, the ultimate disposal of finished products, only a small fraction of the waste material combusted contains carbon fiber composite; in a cubic meter of effluent, only about 10^3-10^5 carbon fibers may be emitted in the presence of about 10^9 or greater particles.

Evaluation of Monitoring Methods

Candidate Methods

Most of the instruments potentially useful for continuous measurement of carbon fiber mass and number were developed in classified government programs to determine fiber release rates in simulated fires and explosions of aircraft. In addition, several methods, developed for other types of fibers, such as asbestos, may be adapted to measure carbon fibers. Fibers from a manufacturing facility differ from those from explosive combustion sources in terms of emission duration, fiber concentra-

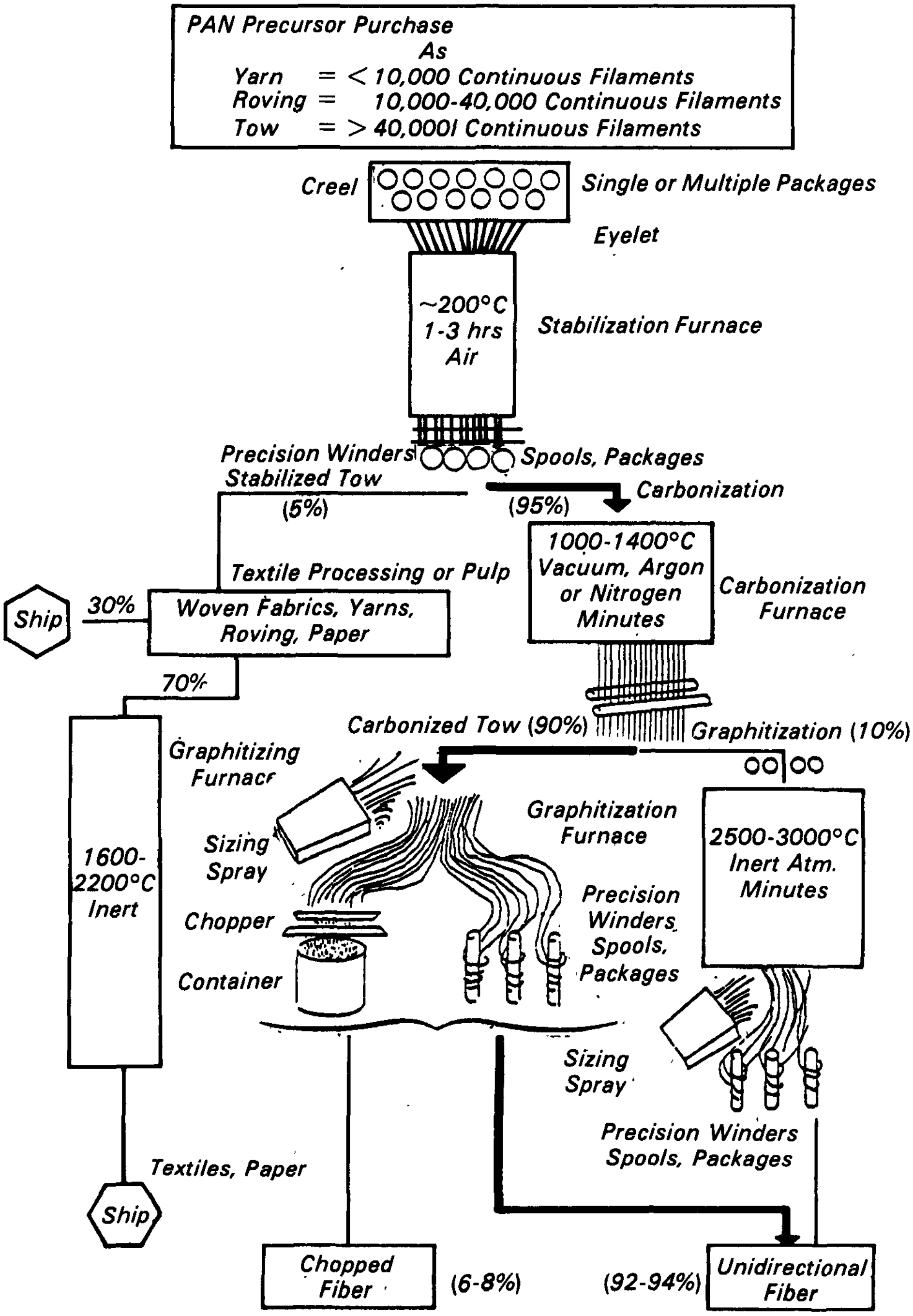

Figure 1. *Carbon fiber manufacturing flow sheet.*

tion and length of fibers. It is possible, however, to extrapolate performance data from previous studies to requirements for a continuous monitor for carbon fibers in the manufacturing environment.

Instrumental methods used for carbon fiber measurement or that have potential as a measurement method are listed in Figure 2. These may be grouped into categories for contacting, locally sensing and remote sensing.

Evaluation Criteria

Various instrumental concepts/devices that have been used or that have potential for carbon fiber measurement were reviewed. Several of these devices are in the conceptual stage only. Some devices, which are commercially available, are applicable to the selective measurement of fibers of any type while other devices permit measurement of total particulate matter which includes fibers as a fraction of the total.

Each device/concept can be evaluated on the basis of several factors which define (1) the analytical adequacy of the instrument, (2) the appropriateness of the device to continuously monitor carbon fibers in a manufacturing environment, (3) the physical practicability of the device and (4) the requirement for improvements to achieve adequate operation. Specific evaluation parameters are documented in Figure 3. Each parameter has been assigned a weighting factor (WF) on the basis of its perceived importance to carbon fiber measurement.

Carbon fiber concentrations may vary markedly depending on intermittent manufacturing processes. Therefore, measuring the concentration over a wide range is very important. The working concentration range, from the detection limit to the saturation point, should be as large as possible. The desirable range, of course, depends on the nature of the environment to be sampled.

The size and shape of the particles are critical. Detecting carbon fibers to the exclusion of all other particles requires that certain size and aspect ratio values be met. Specifically, carbon fibers have lengths ranging from 5 μm to >10 mm and diameters from <1 to 10 μm. The aspect ratio for such fibers may range from 3:1 to 1000:1. It appears that fibers of 1 to 10 mm in length are most hazardous to electrical equipment while smaller fibers are most hazardous to the human respiratory system.

Measurement Parameters

Three possible operating schemes are available. First, a sample may be extracted from the effluent stream and measured in a separate location. This scheme requires equipment (e.g., pumps) to extract a sample and may alter the condition of the sample between effluent duct and measurement point. Second, a device may be installed in the sample line and measure the concentration of carbon fibers as they flow by. This scheme may require periodic cleaning of the sensing device to maintain accuracy. Third, the carbon fibers may be monitored remotely, thus

Table 1. *Carbon Fiber Emission Scenarios**

Parameter	Scenario A	Scenario B	Scenario C
Process	Textile Weaving	CF Composite Machining	Waste Disposal by Incineration
Control Procedure	Room Filtration	Hood/ Baghouse	?
Air Movement			
Gas Volume (m^3/min)	~100	5-20	200-300
Duct cross section(m^2)	~8	0.01-0.1	10-15
Linear velocity (m/sec)	0.2	1-2	5-10
Temperature (°C)	ambient	ambient	100-150
Moisture (%)	-	-	10-20
Total Particulate Mass (mg/m^3)	1	5-10	200-500
Corrosive Gases	no	no	yes
Maximum CF Concentration (number/m^3)	10^7-10^8	10^8-10^{10}	10^3-10^5
Average Length (mm)	5	0.5	1
Length Range (mm)	0.5-25	0.1-2	0.1-10
Presence of			
other particulate	no	yes	yes
other fibers	no	maybe	probably
other conducting fibers	no	no	maybe

Source: Arthur D. Little, Inc., estimates.

[Added Note: A reviewer of this final report brought to our attention the following measured data obtained on EPA Contract No. 68-02-3229.]

	A	B
Total Particulate Mass (mg/m^3)	0.1-10	-
CF Concentration Range (number/m^3)	10^3-$5x10^4$	10^4-10^8
Average Length (mm)	1.5	0.1

precluding any interaction of the instrument with a corrosive medium. The last scheme is ranked as most useful although it is acknowledged that it may be the least suitable on other grounds.

The sample volume is important for two reasons. A larger sample potentially provides a lower detection level and a more representative sample. A sampling interval of small duration is preferred since it provides maximum protection against short term release of environmentally hazardous fibers. Continuous monitoring provides such protection and can yield time-weighted averages of fiber concentration.

Contacting

- *Ball Gauge*
- *Electric Grid - Arc*
- *Electric Grid - Resistance*

Locally Sensing

- *Optical*
  - *Opacity*
  - *Scattering*
    - *Fiber Aerosol Monitor (FAM)*
    - *Near Forward Scattering*
    - *Rotating Lens*
- *Microwave*
  - *Interception*
  - *OSGEF*

Remote Sensing

- *Radar*
- *Infrared*

Figure 2. *Candidate monitor methods.*

Detection Parameters

Selectivity and sensitivity are of critical importance for environments containing carbon fibers, non-conductive fibers and other particles. This may not be necessary in some environments. As noted above, the morphology of carbon fibers, i.e., clumps, bundles, single fibers, is important because of their aerodynamic behavior. Single fibers are generally of most concern. In many cases, fibers must have a specific orientation to be detected. For example, a fiber may pass undetected through an electric grid if it is oriented perpendicular to the plane of the grid or a carbon fiber may not be differentiated from a non-conductive fiber in a light scattering device if it does not rotate in the sensing field. If fibers need to be oriented, more complex instrumentation is required. The simplest devices do not require orientation.

Frequent failure of the instrument to detect carbon fibers increases hazards; frequent false positives increase costs.

Instrument Parameters

The instrument parameters in Figure 3 are self-explanatory. Some parameters, such as size and power require-

Detection Range

Fiber Concentration (fibers/cm^3)	10^{-2} — 1 — 10^2 — 10^4
Fiber Length (cm)	10^{-3} — 10^{-2} — 10^{-1} — 1 — 10
Fiber Aspect Ratio	3 — 5 — 10 — 20 — 50 — 100
	→ Increasing Usefulness →

Evaluation Parameters	WF	Value (1 – 2 – 3 – 4 – 5)	Score
1. Measurement Method			
Sampling	1	*Extract → In-Line → Remote*	
Volume	2	*Small Fraction → Large Fraction*	
Time	1	*Long → Short → Continuous*	
2. Detection			
Selectivity	3	*Particle--------Fibers--------------- CF*	
Basis	2	*Mass-------Number -------------- Both*	
Morphology	1	*Clumps-------Bundles---------Singles*	
Sensitivity	2	*Low ------------------------------ High*	
Need for fiber orientation	.1	*Yes--------By Method -------------- No*	
Detection time	1	*Min----------Sec.---------------- Cont.*	
Frequency of error	2	*Often-------------------------Seldom*	
3. Instrument Parameter			
Physical size (weight)	1	*Large--------------------Small*	
Power requirements	1	*High--------------------- Low*	
Ruggedness	2	*Fragile-------------------Rugged*	
Maintenance	1	*Often--------------------Seldom*	
Calibration	1	*None--------------------Primary*	
Unit cost	2	*High---------------------Low*	
Development cost	1	*High---------------------Low*	

4. Disadvantages (Negative Scores) — *Subtotal* [ ]

Concept only - no experimental verification	(-10)
Based on limited laboratory experiments	(-5)
Other:	( )
i.e., High false positive count in	( )
moist stream	(-3)
Concentration if function of velocity	(-3)

Less [ ]

Total Score [ ]

Figure 3. *Evaluation criteria.*

ments, are dependent on sampling environments. In other cases, (e.g., ruggedness; unit cost) the parameter relates to the usefulness of the device.

Other Considerations

The evaluation criteria include a subjective ranking of deficiencies in sensitivity or selectivity of these methods or concepts for carbon fibers.

Ranking of Methods

Weighting factors for individual parameters were chosen on the expectation that detection parameters are most important followed by measurement parameters and instrument parameters. However, since the weighting factor is critically related to a specific sampling environment, final ranking (based on numerically-weighted scores) of all methods is given on the basis of the three sampling scenarios described above.

Comparison of Methods

Evaluation sheets were prepared for each of the candidate methods listed in Figure 2, following the model presented in Figure 3. Note that the estimated fiber concentration (number) and length ranges for these methods tend to fall into one of three regions:

1. Very high concentrations, long length
   - Optical-opacity
   - Microwave interception
   - Radar
   - Infrared
2. Moderate concentration, long length
   - Ball gauge
   - Electric grid - arc
   - Electric grid - resistance
   - Microwave - OSGEF
3. Moderate concentration, short length
   - Optical scattering

These regions, which do not overlap, are shown in Figure 4 together with the location of the three emission scenarios given in Table 2. Scenarios A and C fall near boundaries of the Region 2 methods. Carbon fibers produced in B would be detected by most of the Region 1 methods; however, these methods measure total particulate and are not specific to carbon fibers, which limit their usefulness to specific applications. Region 2 methods would detect the presence of Scenario B carbon fibers but would underestimate their concentration. Such devices could still be useful for indicating an excursion in carbon fiber concentration above some acceptable level.

The Region 3 optical scattering methods are expected to cover a wide range of concentration but are limited to relatively short fibers. These methods would be the only useful approach for

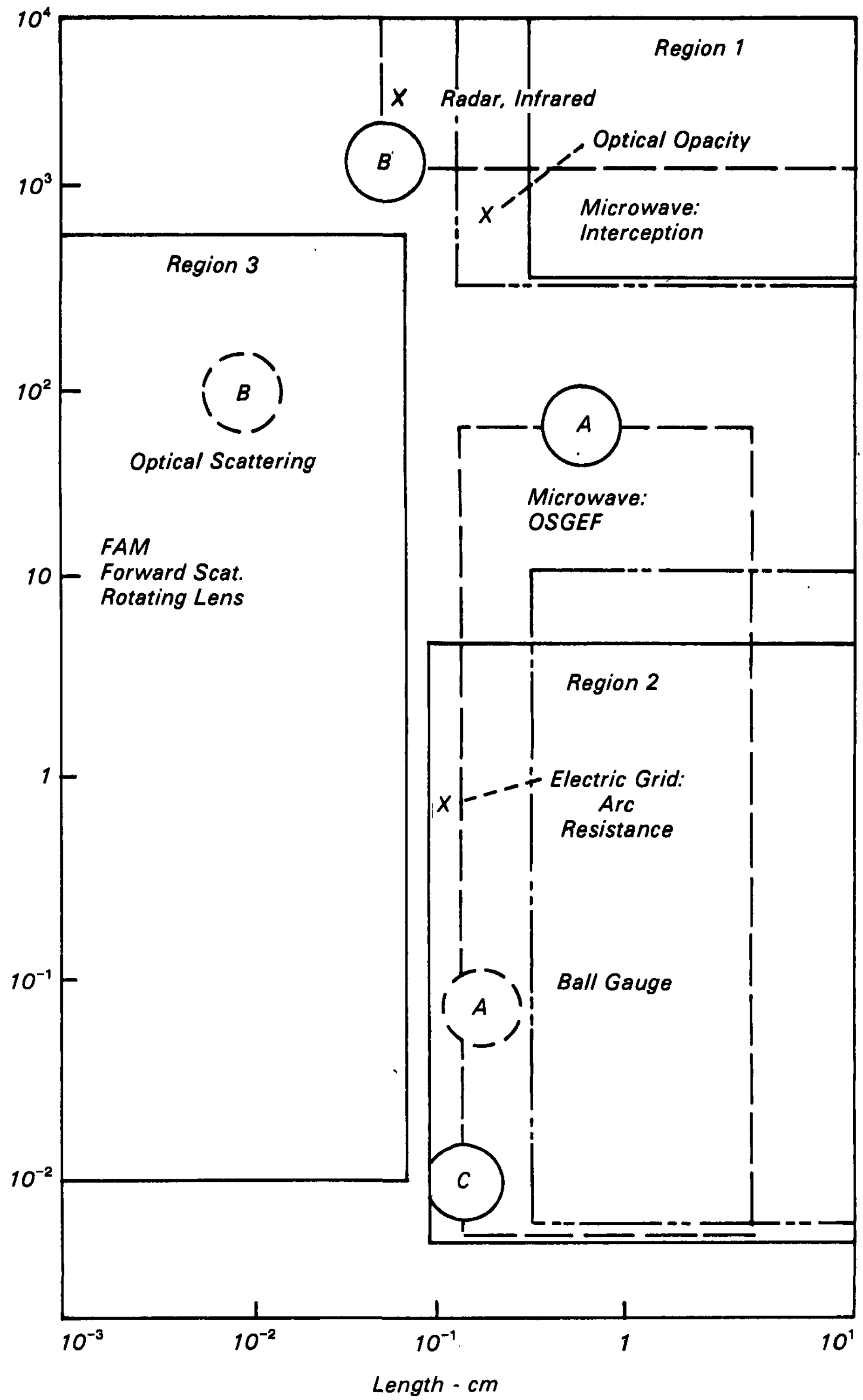

Figure 4. *Detection range of candidate carbon fiber monitor methods. (See Figure 1 for description of scenarios A, B and C.)*

monitoring respirable fiber sizes and could be used in conjunction with one of the Region 2 methods for complete coverage of any length of carbon fiber over three to four decades of concentration.

The methods which are felt to be most promising for application as a carbon fiber monitor are as follows:

1. Fibers: 1 mm and longer in presence of background particulate.

   - *Microwave OSGEF* - The major advantage of the method is the highly specific identification of carbon fibers in the presence of other matter, including conductive particles and fibers. The method could be applied over a large dynamic range of fiber concentration. Further development studies are required to establish detection limits with respect to measurement time, flow rate (volume), minimum length and maximum concentration, the last being influenced mainly by particle coincidence. The detection section of the instrument is simple, and data processing and display can be remote.

   - *Electric Grid - Arc* - The device may be constructed to intercept a major portion of a flowing airstream, thereby providing good representativeness. Detection is based on particle interception between two or more electrodes. Fiber counts can be obtained over a large dynamic range. The method is only approximately length specific, depending on electrode spacing, and fibers suspended parallel to the airstream may be missed. False positive counts are possible depending on the nature (conductivity, size) of other particulate matter. Data processing and display can be remote. Data processing for length and mass concentration may be complex. The instrument may require frequent cleaning and calibration, depending on the nature of other particulate and entrained moisture.

2. Very high concentration of fibers in absence of other particulate matter.

- *Optical-Opacity* - The light-emitting diode (LED) is simple and inexpensive; it is suitable for detecting a very high emission of carbon fibers in the absence of other particulate matter. For example, this device could trigger an alarm in the event of a process upset where a very large number of fibers were emitted and drawn through an exhaust hood duct. The device is limited to a measure of total particulate matter, giving no information about carbon fiber size, number or mass concentration.

3. Respirable fibers.

- *Optical Scattering - Fiber Aerosol Monitor (FAM)* - The usefulness of the FAM instrument has been demonstrated for the measurement of asbestos fibers. With some modifications to increase sampling volume and the length of fibers detected, the instrument could be usefully applied to measure the number and size distribution of carbon fibers. The instrument is complex, expensive and cannot distinguish between fiber type. Based on results for measuring asbestos, the measurement is accurate and precise.

- *Optical Scattering - Rotating Lens* - This method is at the concept stage. Further design and laboratory evaluation is required. The method offers the possibility for measuring a large range of fiber lengths, limited only by particle (or fiber) coincidence. The "sampling" of the stream is remote and can be arranged to traverse across the duct. As with other optical scattering methods, fibers of all types are measured.

Conclusions

Relatively few opportunities for release of carbon fibers occur during the making of carbon fiber epoxy composites (fiber chopping, tow rewinding and textile weaving) and during the final shaping (grinding, sawing and drilling) of products to which the composite has been applied. Local emissions are controlled by exhaust hoods and water sprays. Laboratory simulations of finishing operations show that the majority of fibers released are less than 0.1 mm in length. Three emission scenarios were developed to represent the range of conditions that may be encountered in the manufacturing, application and disposal of carbon fibers. These were textile weaving, carbon fiber composite machining and incineration.

Eleven measurement methods or concepts were identified as candidates for the continuous measurement of carbon fiber emissions. These candidates were scored according to parameters concerned with measurement (sampling), detection, instrumentation and with detectable ranges of fiber concentration and length compared to the three emission scenarios. No single monitoring instrument is suitable for all possible types of emission. Fiber length and concentration ranges must be specified to permit selection of the appropriate instrument.

Monitor methods recommended for further study are:

- Moderate to high concentration, length > 1 mm
  - Microwave OSGEF (Arthur D. Little, Inc.)
  - Electric grid-arc (Bionetics, JPL).
- Moderate to high concentration, length < 1 mm
  - Optical Scattering-rotating lens (Epsilon Laboratories, Inc.)
  - Optical Scattering-FAM (GCA, Inc.).
- Very high concentration, only carbon fibers
  - Optical-LED (commerically available).

Microwave OSGEF is the only method that is specific to carbon fibers. The optical methods detect all fibers, including glass and polymer fibers. The electric grid-arc method measures all conductive fibers (and particles which cross several electrodes) and may be adversely affected by moisture content in the air stream.

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The complete report, entitled "Monitoring Techniques for Carbon Fiber Emissions: Evaluation B," (Order No. PB 81-247 413; Cost: $9.50, subject to change) will be available only from:
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