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PHYSICAL PROPERTIES OF REDISPERSED IRON
FIBERS MEASURED AT 94 GHz

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13. ABSTRACT (Maximum 200 words) Millimeter wavelength attenuative properties of iron fibers produced by the high temperature reduction of iron carbonyl were measured in both controlled and field environments. Aerosol morphology was evaluated statistically for the controlled studies, and results were used as a basis for calculations of the absorption efficiency. Scattering efficiency for such small diameter fibers can be assumed to be a negligible proportion of the net attenuation. Reasonable agreement between measurements and theory is indicated. Efficiencies remain relatively high for visible wavelengths.				
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1. Introduction

This report gives results of laboratory and field measurements of the millimeter wavelength optical properties of iron fibers at 94 GHz. These fibers are produced by the high temperature reduction of $\text{Fe}(\text{CO})_5$ to iron condensate spherules (diameter $\approx 0.1 \mu\text{m}$) which coalesce in the final stages of their growth to form chains or fibers. In the presence of a magnetic field, they become quite straight; otherwise they would closely resemble, for a common example, carbonaceous soot. Examples of the iron fibers are shown in figure 1.*

The laboratory measurements were performed under carefully controlled conditions using redispersed fibers. Since the laboratory aerosol was well characterized, one can estimate the optical result using either the ellipsoidal theory or a much more precise theory by Pedersen et al. (1985, 1987) and Waterman and Pedersen (1992). The agreement for the latter was well within the limits of the uncertainty and suggested the potential for very high extinction (absorption) efficiency for longer fibers. However, it has not been established that the longer iron fibers have the strength to withstand redispersal.

Field measurements of iron fibers generated in-situ are also included and remind the reader of another feature predicted by the theory (Waterman and Pedersen 1992, van de Hulst 1957) for this aerosol; that is, the extinction efficiencies at the short wavelengths (visible, infrared (IR)) are even higher than those for the millimeter wavelengths. This, of course, is reversed for the 3- to 10- μm diameter graphitic particles (Bruce et al. 1990a, 1990b). Unfortunately, because of inadequate characterization (sample oxidized on the substrates) and particle geometrical complexity (multiple connectedness, nesting) for these iron fiber experiments (1984), meaningful comparison with the theory is not feasible.

2. Definitions

Since the research of this group involves both of the components of the extinction, the usual definition of small alpha for the latter quantity is inconvenient. Therefore, the symbols will be defined as follows. T , the transmissivity over the pathlength l is defined by

$$T = e^{-\int \kappa dl}$$

*Figures are located at the end of the report.

where

$$\epsilon = \rho \epsilon$$

Small epsilon, $\epsilon(\text{m}^2/\text{g})$, is the extinction efficiency defined as the extinction coefficient, $\epsilon(\text{m}^{-1})$, divided by the mass density, $\rho(\text{g}/\text{m}^3)$, of the aerosol in a volume of space. Then,

$$\epsilon = \alpha + s_t$$

where α and s_t are the absorption and total scattering efficiencies.

3. Laboratory Measurements

A magnetic "fluidizer" was constructed by Rutstein Associates, Oberlin, Ohio, to disperse the iron fibers. This unit was extensively modified to supply solely single fibers in a gentle airflow pattern. The functional system is described in three illustrations (figures 2, 3, and 4) and their captions. The last of these figures shows the sampling systems that tap into the airflow of the fluidizer duct (10 cm diameter tube). Note that the photoacoustical and an aerosol characterization system are in series; that is, the same sample is optically measured and then collected for analysis.

The photoacoustical system was first described in Applied Optics in 1984 (Bruce and Richardson 1984) and more recently in 1991, in the same journal (Bruce et al. 1991). Photoacoustics, which measures only the absorption, is appropriate in the case of these short ($L \ll \lambda$), small-diameter high-conductivity fibers. Scattering will account for well under 1 percent of the extinction.

The 94 GHz absorption efficiency, measured photoacoustically in the laboratory, for separated fibers, using gentle processing in the magnetic fluidizer was

$$\alpha = 2.2 \times 10^{-1} \text{ m}^2/\text{g} \text{ (1987) ,}$$

for a length distribution peaked near 70 μm .

Another value was obtained by using a somewhat stronger fluidizer oscillating magnetic field. In this case, multiple particles were more often obtained. These particles would often partly overlap, effectively creating longer particles. Theoretically, the efficiency increases in a power-law relationship with length, $L^{3.49}$, in the "Rayleigh" region

(includes all the fibers measured) while the power-law relation for the diameter is weaker $\sim d^{-2.53}$, in the opposite direction with respect to the efficiency. Thus, a measured increase to $\alpha = 4.1 \times 10^{-1} \text{ m}^2/\text{g}$ for the somewhat siamesed particles is not a surprise.

Precise comparison with the strong power law functions for the diameter and length of the theory is difficult for fibers with $kL \ll 1$, particularly for the diameters (k is the propagation vector). As the photographs of figure 1 show, the effective particle diameter is often not well defined, being complicated by the general "lumpiness." These fibers, like all condensate solid aerosols we have seen, started growth as spheres and coalesced when their radii were of the order of tens of nanometers. Growth then continued in this case to a mean diameter of the order of $\sim 0.2 \mu\text{m}$.

The calculational estimate was based on measured length (detailed) and diameter (approximate) distributions. Graphs of the distribution of lengths and the consequent calculated optical efficiencies are shown in figures 5 and 6.

Functional dependencies were taken from calculations based on Pedersen et al. (1985, 1987) and Waterman and Pedersen (1992). The result for the separated fibers was

$$\alpha = 1.12 \times 10^{-1} \text{ m}^2/\text{g}.$$

Here we remind the reader that the fibers measured were separated about as gently as is possible. The system airflows were carefully managed for this (relatively low velocity and turbulence). As figure 4 shows, all measurements were made in series (photoacoustic first, then dosage and sizing from the same sample). It is not likely that longer single fibers could be extracted from the nested sample provided. Therefore, the values are probably near maximum for that material.

When the variation in diameter along the fibers is considered, the agreement between measurement and theory is quite good. Samples with longer separable fibers would provide another point validation.

4. Comments

Given the strong power-law relationships for optical efficiency functions of the two basic fiber dimensions, an increase in separable fiber length for the redispersed material would be valuable for testing the theory and should be sought. However, an altered process of generation leading to a decrease in fiber diameter of more than about a factor of 2 is unlikely. Fiber strength becomes a consideration for longer lengths and smaller diameters. At some point the joints would become too weak to allow collection and redispersion. (Soot "fibers" often have diameters of less than $0.1 \mu\text{m}$ but are very fragile.)

Even a factor of 2 decrease in diameter would increase the efficiency by about a factor of 6, according to the theory: not enough . . . by itself. Fiber length, then, is the primary candidate parameter for improvement. Theory says that simply increasing the length by, for example, a factor of 3 yields an efficiency increase of about 50! If this length is difficult to attain, then consider an increase of only a factor of 2, which gives an efficiency improvement of 11.

The dimensional and thus optical efficiency limits for the redispersed material are therefore indirectly related to the strength of the material and the magnetic forces between adjacent fibers as they nest. This could be calculated without great complication. A small contract to quantify the dimensions for maximum optical efficiency with the bounds of material strength for ferromagnetic forces would be the quickest way to determine the potential of these fibers.

5. Field Measurements

There is an additional item of data that relates to the efficiency of this material. Measurements during a 1984 field experiment at both 140 GHz ($\lambda = 2.14$ mm) and $\lambda = 0.63$ μ m gave the absorption/extinction efficiencies listed below. If the diameters are assumed to be the same as those measured in the laboratory for the study of this report, the Pedersens/Waterman theory would have it that the once-dispersed fibers of the field test were effectively only about 40 percent longer than those of our laboratory measurements (recall that they are multiply connected). The implication is that, assuming similar particles for the two situations, the fluidizer caused comparatively little breakage. In any case, the generators were not producing particles that were long enough to realize a significant portion of the iron fiber potential.

Extinction/absorption efficiencies (m^2/g) measured during field experiments of 1984.

	<u>140 GHz</u>	<u>0.63 μm</u>
Run 1	0.67	3.2
Run 2	0.79	4.2

Finally it should be noted from the above data that this would be a truly broadband absorber if the length could be increased as discussed above. This is not true for the fibers of the other category (chopped extruded fibers, Bruce et al. 1990) as their diameters preclude satisfactory efficiency in the IR.

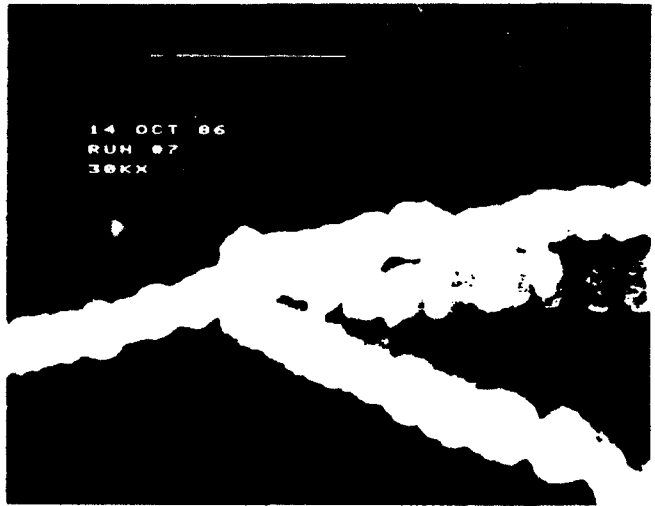
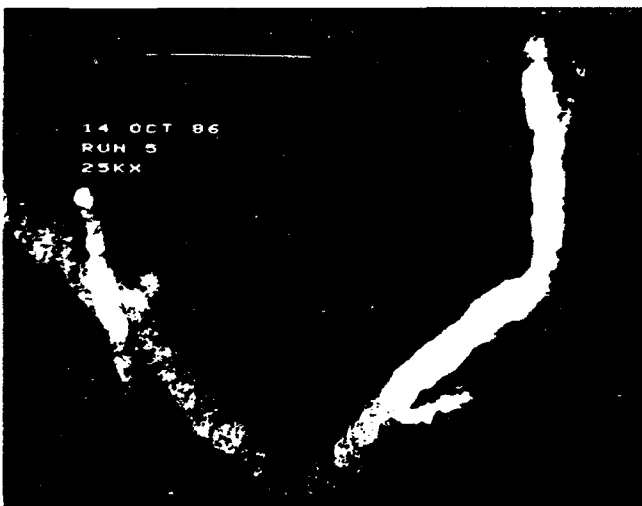
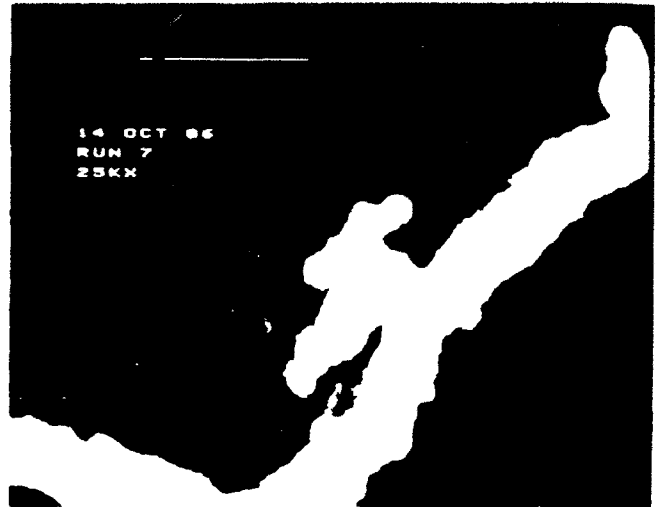
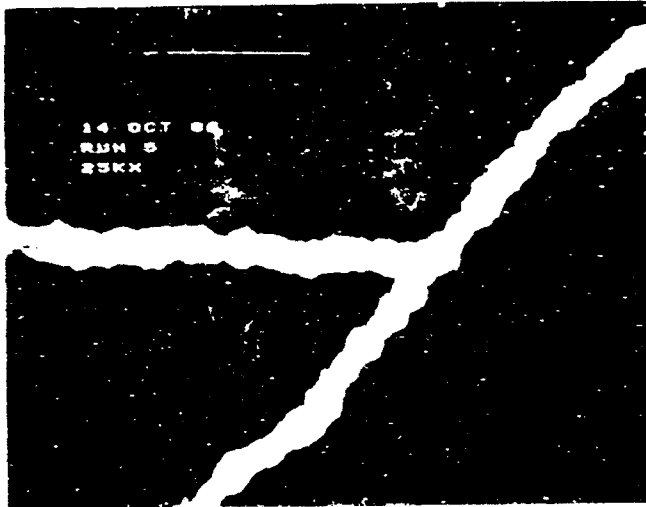
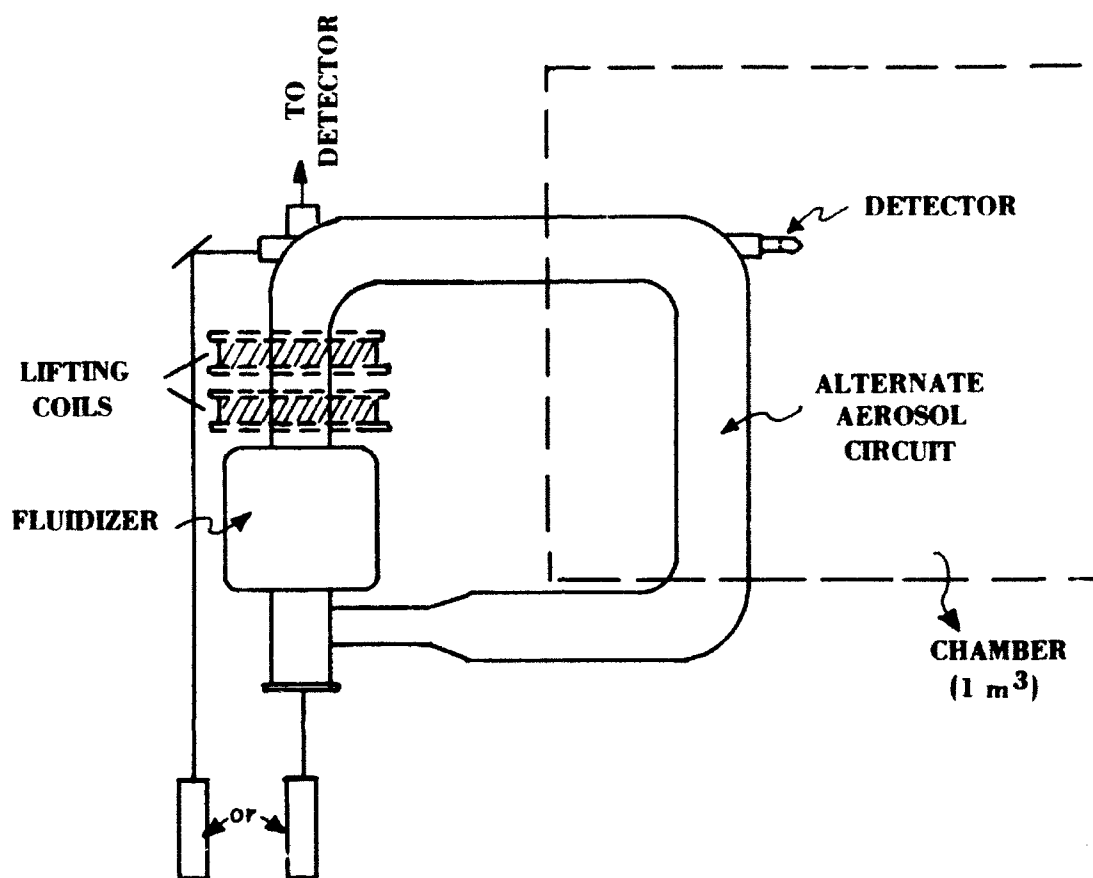


Figure 1. Scanning electron micrographs of the iron condensation particles.

**SIDE VIEW: FLUIDIZER SYSTEM
AND FLUIDIZER DIAGNOSTICS**



HeNe PROBE SYSTEMS

Figure 2. Fluidizer and airflow circuit. During the laboratory study, the smaller volume (alternate) flow circuit was used. A nonintrusive flow mechanism (Alprin ejector) was installed in series in the 10-cm-diameter tubing. A sketch of the removable section at the base of the fluidizer is shown in figure 3.

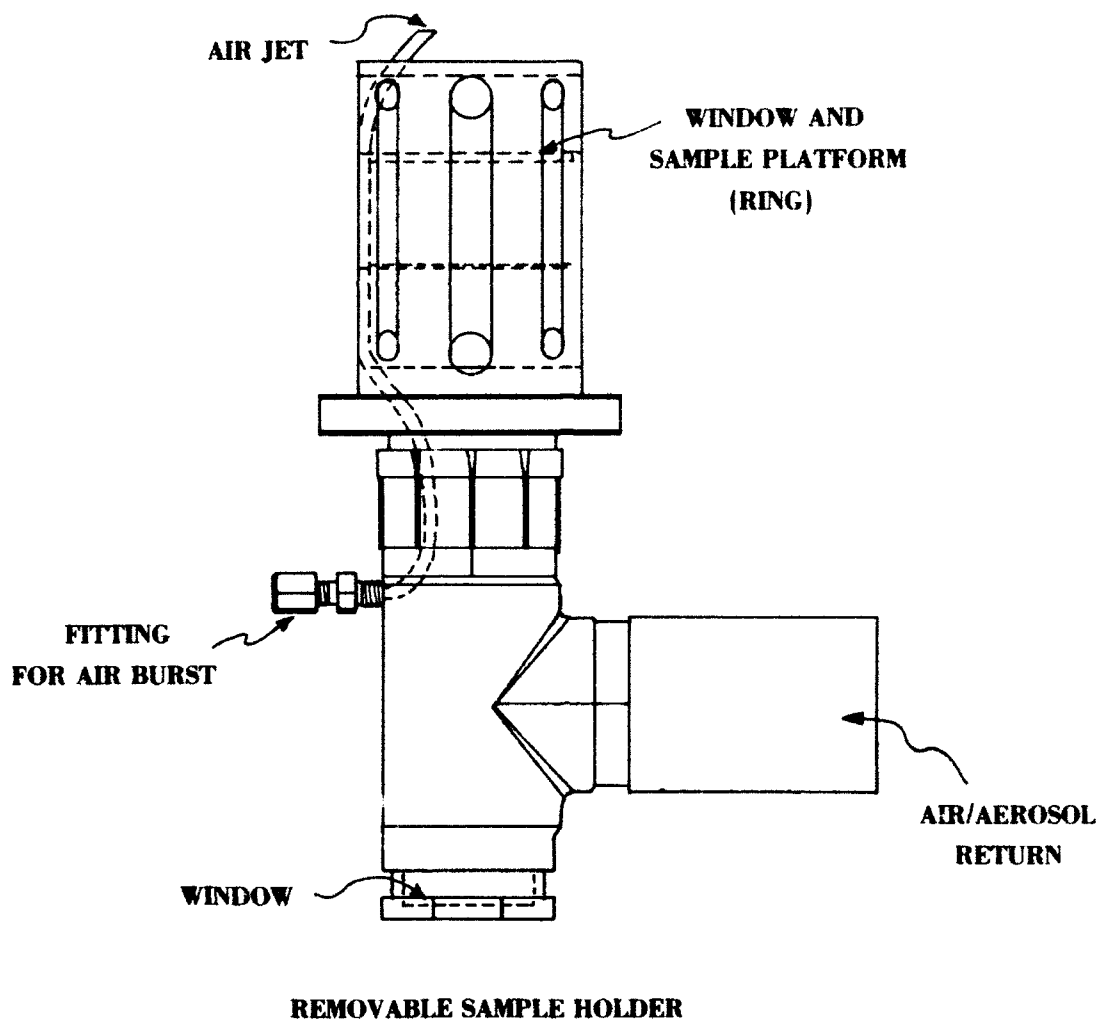
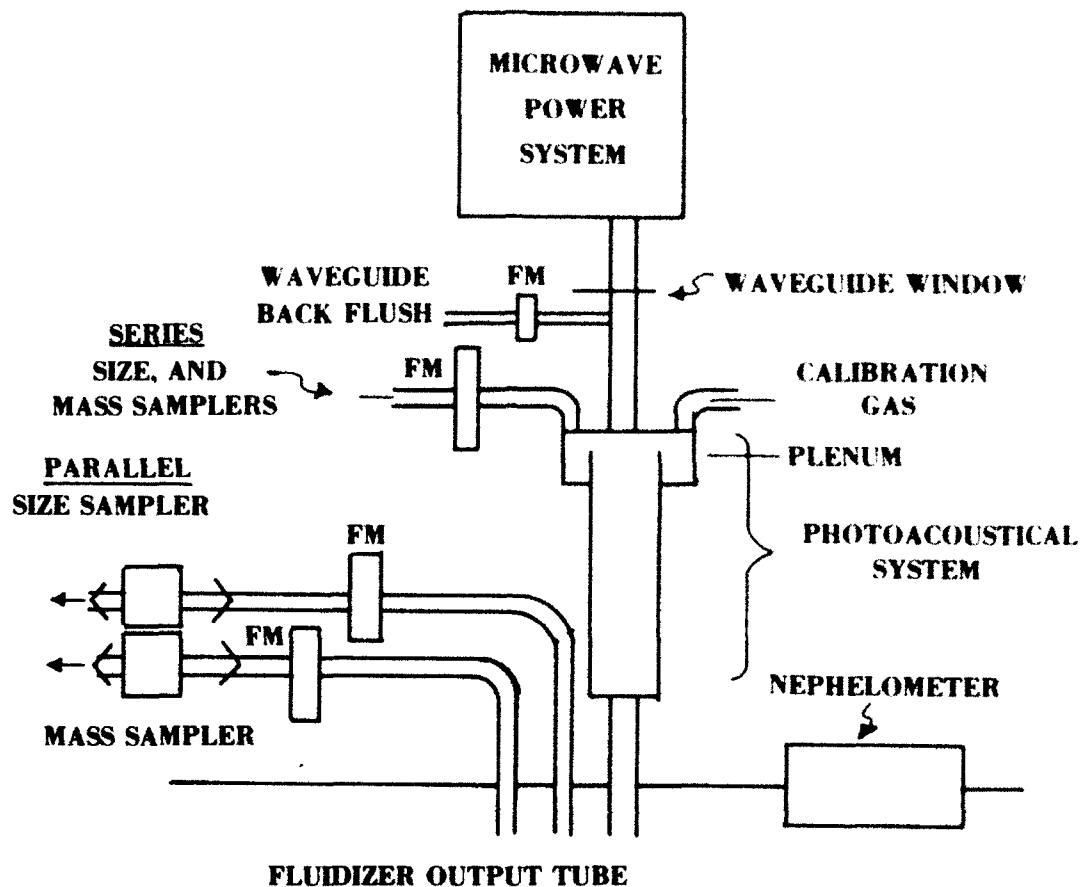


Figure 3. Removable section at base of fluidizer. This unit has several important functions. The air/aerosol returns to a plenum just underneath the glass sample holder and passes along channels that enter the fluidizer chamber above the sample platform. A glass window at the base of the plenum allows the laser probe beam to pass through the center of the fluidizer chamber after the particles have been levitated. There is also an air jet near the center of the chamber that can be used to provide bursts of air when the material is too nested to be separated by the fluidizer.

TOP VIEW: SAMPLING SYSTEMS



... FM = FLOWMETER

Figure 4. Microwave and aerosol characterization system. The type of microwave photoacoustical system (spectrophone) used was described some time ago by Bruce and Richardson (1984). Aerosol characterization systems were arranged in both parallel and series sampling circuits with respect to the spectrophone. The assurance of characterizing the identical sample measured photoacoustically was too strong an approach to ignore. A nephelometer operating in the near IR, sampling with the primary flow circuit, served as a reference aerosol density detector for fluidizer performance. Another nephelometer (shown) was installed in series with the spectrophone for correlation of the time dependence. Dosimetric samples were obtained on polycarbonate filters and were analyzed using atomic absorption spectroscopy (AAS). Size analyses were obtained using the same filters before the destructive AAS process. All flowmeters were calibrated using menisci of soapy water moving in a glass tube.

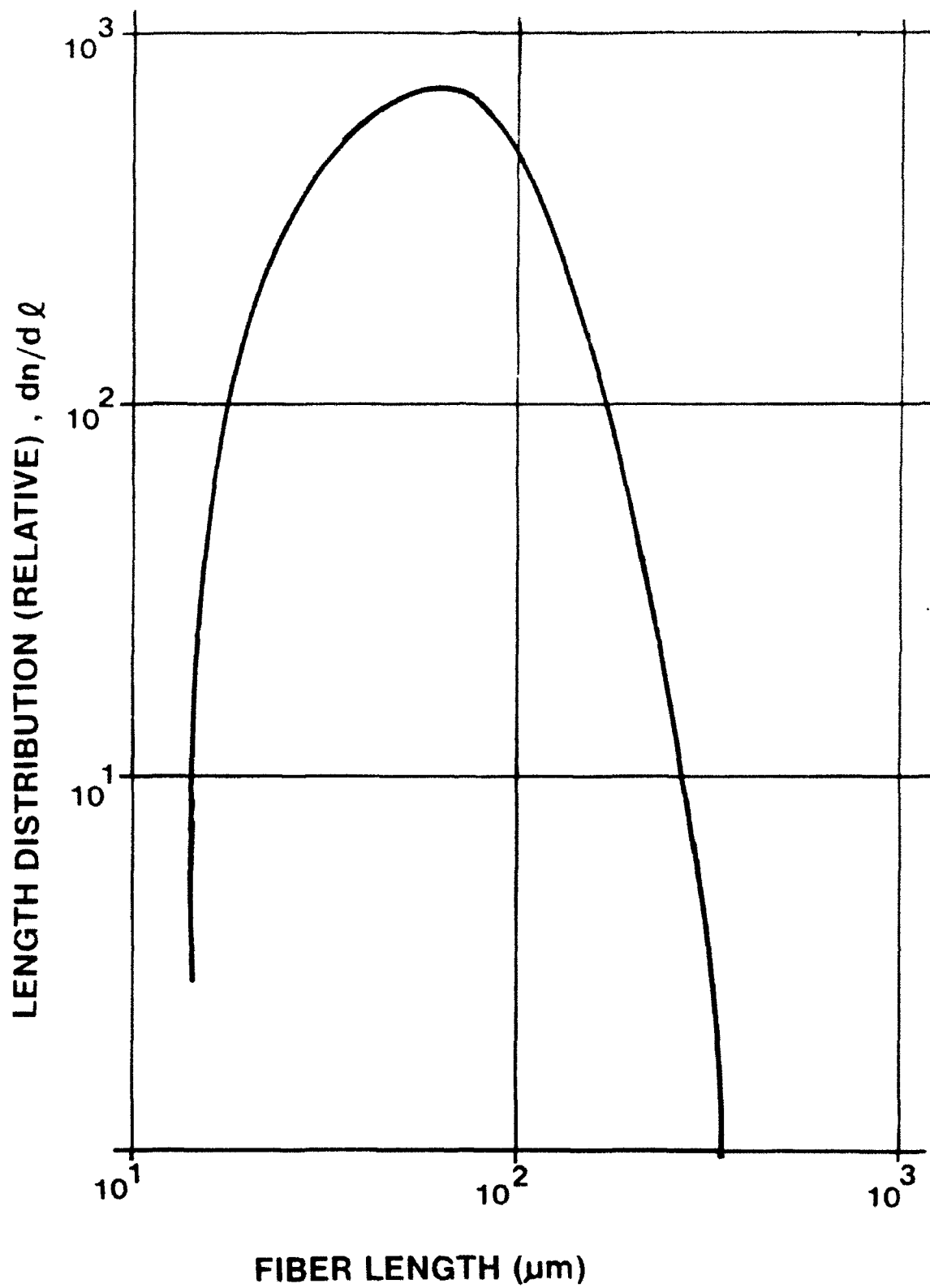


Figure 5 . Single fiber length distribution.

RELATIVE DIFFERENTIAL ABSORPTION COEFFICIENT

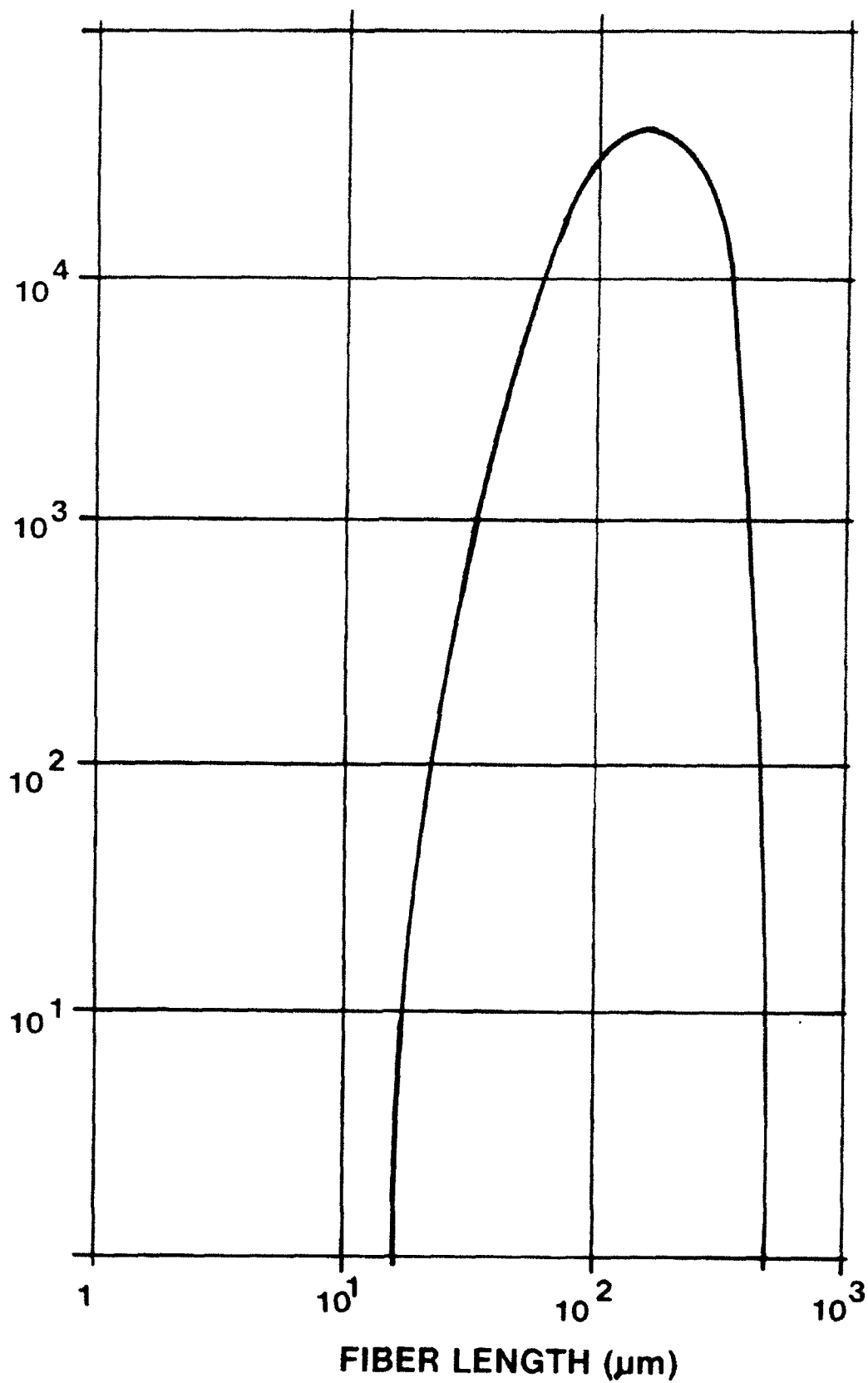


Figure 6. Calculated extinction, absorption with length.

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