CHARACTERIZATION OF COAL MINE DUST BY COMPUTER PROCESSING OF SCANNING ELECTRON MICROSCOPE INFORMATION *

E. W. White

Department of Material Science
The Pennsylvania State University
University Park, Pennsylvania 16802

P. B. DeNee

Pittsburgh Mining and Safety Research Center
Bureau of Mines, United States Department of the Interior
Pittsburgh, Pennsylvania 15213

INTRODUCTION

The Coal Mine Health and Safety Act of 1969 has set an upper limit on the average concentration of coal mine dust at three milligrams per cubic meter of air. This limit was to be enforced as of 1970 and is to be reduced to two milligrams per cubic meter of air by 1972. In order to assist mining companies in meeting this goal, the United States Bureau of Mines was assigned the responsibility for designing and implementing a research and development program to provide the advanced technology for reducing respirable coal mine dust. The present paper reports on a portion of this program. It is specifically involved with the sampling, identification, and characterization of the respirable dust, both in the experimental laboratory system and in working coal mines. The characterization of dust is necessary to understand the aerodynamic behavior of the dust, to identify its origin, and to monitor the effectiveness of dust suppression techniques.

Respirable coal mine dust is petrographically quite complex, consisting of fragments of the various coal macerals as well as mineral and rock fragments; it is therefore of interest to include the size and shape of each type of particle in the characterization. The various components or particle types also quite likely behave differently as aerosols, even when the particles have a common size and shape. In addition, the evidence in the literature [1, 2] indicates that there probably are differences in the size distribution of coal particles as compared with the noncoal dust or mineral fraction. It is thus important to include particle type (or elemental composition of the individual particles) in the characterization.

Several systems are being used at the Bureau of Mines for dust characterization, one of the more important being the Scanning Electron Microscope (SEM). The importance of the SEM lies in the fact that individual particles in the respirable size range (0.1 to 10 μm) can be studied with respect to size, shape, volume, surface topography, agglomeration behavior, and elemental composition (leading to particle type),[3, 4] but it is a time-consuming process. In order to draw statistically meaningful conclusions, it is necessary to measure

---

* This research was supported by the United States Bureau of Mines, under Contract No. G0101720 (MIN-40).

a large number of particles. For simple size and shape characterization, a sample of at least 1000 particles is recommended. For size and shape analysis according to particle type, several thousand particles might have to be measured. For these reasons, it was deemed necessary to utilize the automatic Computer Evaluation Scanning Electron Microscope Images (CESEMI)[5,6] techniques being developed at the Pennsylvania State University.

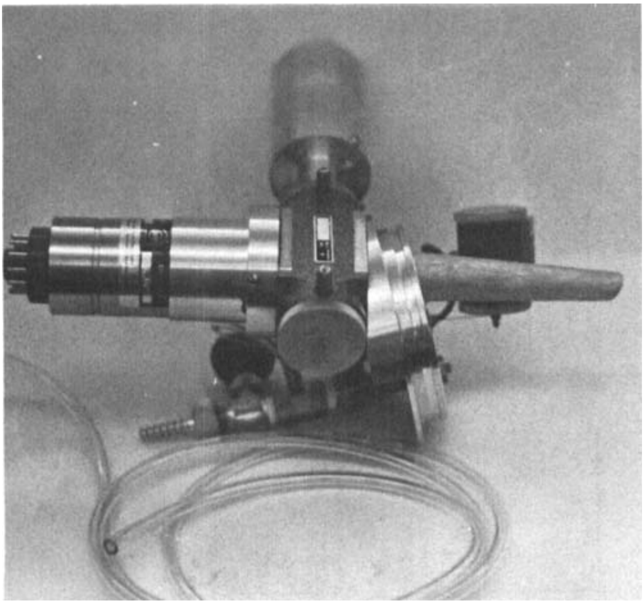

FIGURE 1. Photograph of the three x-ray detectors.

INSTRUMENTATION

The CESEMI technique consists of recording the x-ray and electron information generated by the SEM and later processing this information on a high speed digital computer. The SEM used in this work is a Japanese Electron Optical,* model JSM. The general schematic arrangement is shown in FIGURE 1. (Only one x-ray detector is shown for the sake of simplicity.)

The output of the secondary electron detector is an analog signal that generally varies between about —3 to +2 volts. This signal is fed, in parallel, to the CRT of the SEM and to the signal preprocessor. The preprocessor module was especially designed to manipulate the secondary electron signal. It consists of a bias-level adjustment, an amplifier with a gain continuously adjustable from 1 to 10, and a time constant continuously variable from 0 to

---

* Reference by trade name is only for identification and does not imply endorsement by the Bureau of Mines.

10 milliseconds. The output is continuously monitored on a Tektronix oscilloscope.

An array of three x-ray detectors has been optimized for the simultaneous detection of C, Si, S, Fe, and (Ca + K). The K series x-ray lines are used in each case. A primary consideration in the design was the maximization of x-ray count rate in order to render the analysis in the shortest possible time per particle. This detector array performs at a count rate 80–200 times higher than that obtained by the solid state lithium-drifted silicon detectors commonly used in SEM instruments. The higher count rate was achieved at the expense of detector resolution, which was poorer by a factor of 2 or 3.

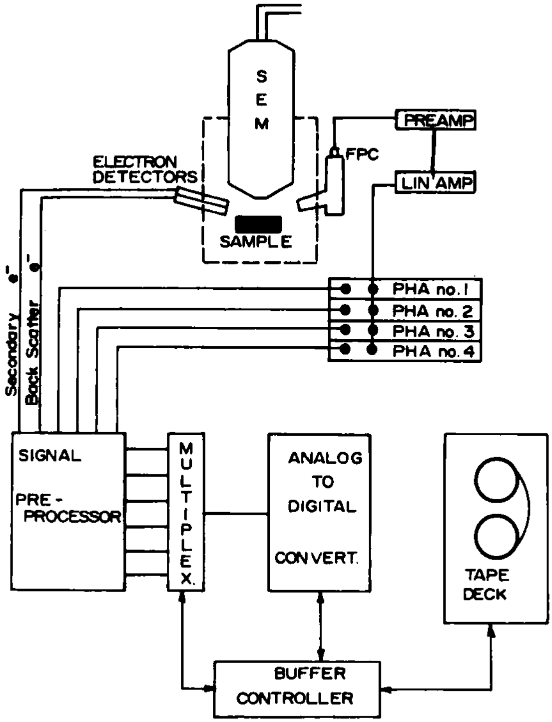

FIGURE 2. Schematic block diagram of magnetic tape recording system.

The detector array, consisting of two flow proportional counters (P-10 gas) and a scintillation detector, is shown in FIGURE 2. The large flow counter is mounted outside the SEM specimen chamber. The collimator allows only x-rays emerging from the sample to reach the detector. This detector is used to pick up the Si, S, and (Ca + K) signals. The scintillation counter mounted in tandem with the flow counter is especially sensitive to Fe Kα. Lighter element radiation is effectively filtered in passing through the flow counter. Carbon x-rays are detected by the third detector, a miniature flow counter with a thin window. The plumbing on the outside of the flange is necessary for the reduced gas pressure operation of the carbon Kα detector.

The signal from the secondary electron detector, along with five x-ray signals from the three x-ray detectors, is processed in the system, and the resultant intensities are recorded by the magnetic tape deck. Each picture point on the image thus consists of the six signals and is typically sampled on a 256 × 256 picture point grid. The available grey scale sensitivity is 1/2000. Details of the instrumentation and the recording procedure have been published previously.[5, 6]

SPECIMEN PREPARATION

Two problems exist in the preparation of coal mine dust samples for SEM characterization. The first is proper dispersal of the particles and more or less uniform distribution on a featureless substrate. The second problem is the nature of the substrate: it must consist of an electrically conductive material that is free of any elements that would interfere with the x-ray detection of the elements of interest.

For best resolution, coal dust particles should be uniformly dispersed on a featureless background without any residue caused by evaporation of the dispersing agent. In this study, three methods of dispersing the dust have been explored. The first involves the direct capture of the dust on the substrate material in a thermal precipitator.[3] The dust thus captured should truly represent the dust as it exists in the air (in the coal mine). The other two methods involve the redispersion of previously captured dust. The first consists of the aspiration of the dust, which is suspended in a liquid such as ethanol,[3] directly on the substrate. The main advantage of this method is that it is quick, since the dust is usually suspended in such a liquid in the process of the removal from the filters or in the capture process itself. The main disadvantage is the lack of control over the amount of dust being deposited.

The last method consists of mixing the precollected dry dust with a eutectic composition of camphor and naphthalene. The mixture is blended and a portion of it is transferred to the substrate, where it is completely sublimed in a vacuum chamber. The eutectic composition (60 wt % camphor/40 wt % napthalene) has dual advantages in that it transforms from a solid to a viscous liquid at a conveniently low temperature (32° C = 89.6° F), and it completely sublimes in modest vacuum, leaving no residue to interfere with the SEM images. This method has the advantage of being the more effective way to deagglomerate the clusters of particles, thus allowing analysis of the individual particulates.

If only size and shape analyses are to be performed on the particles, any smooth substrate such as gold-coated glass can be used. If noncarbon elemental analysis is to be added, cellophane tape can be used as the substrate. Finally, for elemental analysis including carbon, a less conventional substrate must be used, one that does not contribute to the x-ray spectrum in the range of about 1.0 to 50 Å. This restriction limits the choice to substrates consisting of very low atomic number elements. Beryllium has been used, but it is considered hazardous from a health standpoint; it also generally contains a large number of pores that trap polishing material, making cleaning and reuse difficult. It has been decided to evaluate the use of LiF single-crystal substrates coated with boron. The LiF is available as high-density (no pores), single crystals, and boron can be sputtered on the LiF surface to form a hard, electrically conductive coat. The boron can be applied to form a layer just thick enough to absorb the fluorine K radiation. A sputter target of boron has been ordered for this purpose.

COMPUTER ANALYSIS

The recorded images are processed by an IBM 360/67 computer to measure the characteristics of each particle. The description of each particle includes the following parameters:

1. *area*—computed as projected area covered in $(\mu m)^2$, regardless of shape complexity;
2. *perimeter*—length of actual particle perimeter in μm;
3. *ellipse fit*—a least squares fitted ellipse is applied to the particle perimeter, and the properties of the ellipse are computed to give:

   a = major axis dimensions in μm

   b = minor axis dimensions in μm;

4. *particle type*—x-ray information for the five elements is examined by the computer to establish what elements or combinations of elements are present as major constituents. Each combination is stated as a number code. This coding is shown in TABLE 1. Only the eight most probable combinations are included in the table.

TABLE 1

PARTICLE TYPE CODING

	Secondary Electron	Carbon	Silicon	Sulfur	Iron	Calcium and/or Potassium	Code
Coal (carbon)	x	x					16
Quartz (rock) kaolin group	x		x				8
Calcite, dolomite (rock dust)	x	x				x	17
Gypsum, anhydrite (rock dust)	x			x		x	3
Shale, feldspar	x		x			x	9
Pyrite, marcasite	x			x	x		6
Hematite, magnetite	x				x		4
Siderite	x	x			x		20

TABLE 2

EXAMPLE OF COMPUTER OUTPUT FOR COAL DUST SAMPLES

AREA μ^2	E. C. DIA μ	PERIM μ	MAJOR μ	MINOR μ	THETA	ELLIP. AREA	ELLIP. PERIM	X	Y	MINOR/MAJOR	PART. TYPE
8.89	3.36	6.67	0.0	0.0	0.0	0.0	0.0	7	3	0.0	0
12.44	3.98	8.00	0.0	0.0	0.0	0.0	0.0	9	157	0.0	0
5.33	2.61	4.00	0.0	0.0	0.0	0.0	0.0	9	197	0.0	0
12.44	3.98	8.00	0.0	0.0	0.0	0.0	0.0	13	33	0.0	0
32.00	6.38	18.67	0.0	0.0	0.0	0.0	0.0	14	39	0.0	0
5.33	2.61	4.00	0.0	0.0	0.0	0.0	0.0	14	133	0.0	0
23.11	5.42	16.00	4.92	0.93	19.17	14.42	22.26	15	108	0.189	0
12.44	3.98	9.33	0.0	0.0	0.0	0.0	0.0	17	54	0.0	0
7.11	3.01	5.33	0.0	0.0	0.0	0.0	0.0	17	120	0.0	0
5.33	2.61	4.00	0.0	0.0	0.0	0.0	0.0	18	104	0.0	0
21.33	5.21	12.00	0.0	0.0	0.0	0.0	0.0	19	17	0.0	0
3.56	2.13	2.67	0.0	0.0	0.0	0.0	0.0	20	241	0.0	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	21	10	0.0	0
17.78	4.76	12.00	0.0	0.0	0.0	0.0	0.0	21	137	0.0	0
8.89	3.36	6.67	0.0	0.0	0.0	0.0	0.0	26	109	0.0	0
3.56	2.13	2.67	0.0	0.0	0.0	0.0	0.0	26	185	0.0	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	27	23	0.0	0
5.33	2.61	4.00	0.0	0.0	0.0	0.0	0.0	27	101	0.0	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	27	106	0.0	0
206.21	16.20	53.33	10.69	5.31	-44.72	178.27	53.02	27	164	0.497	4
391.09	22.31	106.66	15.44	7.19	-25.70	348.60	75.68	27	221	0.465	0
17.78	4.76	10.67	0.0	0.0	0.0	0.0	0.0	28	38	0.0	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	28	60	0.0	0
110.22	11.85	46.67	11.63	6.25	4.87	228.26	58.66	28	192	0.537	0
7.11	3.01	5.33	0.0	0.0	0.0	0.0	0.0	29	51	0.0	0
19.55	4.99	13.33	3.34	0.96	20.72	10.04	15.44	31	112	0.287	0
7.11	3.01	5.33	0.0	0.0	0.0	0.0	0.0	32	165	0.0	0
110.22	11.85	46.67	15.01	3.14	12.18	147.93	68.14	34	11	0.209	0
74.66	9.75	40.00	10.67	2.58	5.90	86.49	48.79	34	124	0.242	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	34	210	0.0	0
7.11	3.01	5.33	0.0	0.0	0.0	0.0	0.0	34	221	0.0	0
14.22	4.26	9.33	0.0	0.0	0.0	0.0	0.0	37	39	0.0	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	40	191	0.0	0
3.56	2.13	2.67	0.0	0.0	0.0	0.0	0.0	41	2	0.0	0
3.56	2.13	2.67	0.0	0.0	0.0	0.0	0.0	42	162	0.0	0
7.11	3.01	5.33	0.0	0.0	0.0	0.0	0.0	44	43	0.0	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	44	84	0.0	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	44	109	0.0	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	44	172	0.0	0
12.44	3.98	8.00	0.0	0.0	0.0	0.0	0.0	45	23	0.0	0
5.33	2.61	4.00	0.0	0.0	0.0	0.0	0.0	45	168	0.0	0
12.44	3.98	9.33	0.0	0.0	0.0	0.0	0.0	46	176	0.0	0
3.56	2.13	2.67	0.0	0.0	0.0	0.0	0.0	46	213	0.0	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	47	111	0.0	0
80.00	10.09	41.33	18.57	3.30	1.95	192.35	83.79	47	210	0.178	0
7.11	3.01	5.33	0.0	0.0	0.0	0.0	0.0	49	119	0.0	0
5.33	2.61	4.00	0.0	0.0	0.0	0.0	0.0	51	223	0.0	0
28.44	6.02	16.00	3.90	1.57	25.30	19.29	18.68	52	185	0.404	0
1.78	1.50	1.33	0.0	0.0	0.0	0.0	0.0	56	202	0.0	0
517.31	25.66	121.33	0.0	0.0	0.0	0.0	0.0	57	149	0.0	0
3.56	2.13	2.67	0.0	0.0	0.0	0.0	0.0	58	75	0.0	0
426.64	23.31	97.33	14.56	8.18	17.13	374.10	74.19	59	89	0.562	0
28.44	6.02	17.33	4.22	1.88	31.58	24.89	20.53	60	127	0.444	0
280.87	18.91	77.33	16.95	5.46	-9.15	290.83	79.10	61	20	0.322	0

RESULTS AND DISCUSSIONS

Three coal mine dust samples have been analyzed so far. T-30 was a pulverized coal dust sample, Straight Creek Seam, Bell County, Kentucky, and T-31 and T-36 were airborne coal mine dust samples that had been collected using personal sampling filters and ultrasonically stripped from these filters. T-31 was from the Sewell Seam, Nicholas County, West Virginia, and T-36 was from the Lower Kittanning Seam, Clearfield County, Pa. They were redispersed, using the camphor-naphthalene technique on lithium fluoride substrate. The sputtered boron coating was not available at the time of the analyses, so the LiF was coated with a 100 Å layer of carbon to achieve surface conductivity.

An example of the computer printout is shown in TABLE 2; it is interpreted as follows:

1. *Area* = particle area in (μm)^2 computed regardless of shape complexity of the particle. This value is for the projected outline of the particles.
2. *E.C. Dia* = equivalent circular diameter (μm)^2 computed for the area, assuming that the projected outline is circular in shape.
3. *Perim* = the particle perimeter (μm).
4. *Major* = major axis dimensions (μm) fitted ellipse.
5. *Minor* = minor axis dimensions (μm) of fitted ellipse. An ellipse is fitted only if the particle area consists of eight or more picture points; therefore, most of the particles are assigned a value of zero.
6. *Theta* = orientation (degrees) of major ellipse axis measured with respect to the direction of the horizontal raster line. A major axis aligned parallel to the raster line direction has a theta of zero.
7. *Ellip. Area* = area of fitted ellipse computed from the dimensions of the major and minor axes.
8. *Ellip. Perim* = computed perimeter of fitted ellipse.
9. *x* = raster line on which particle is found.
10. *y* = raster column on which particle is found. A value of $x = 7$, $y = 3$, for example, would designate a particle on the seventh raster line at the third picture point along the line.
11. *Minor/Major* = aspect ratio or shape factor of the particle.
12. *Part. Type* = This is the particle-type identification code (see TABLE 1) that indicates the chemical identification based on the combined x-ray information on the particle. In this case, only one high-ash particle was encountered, as indicated by the number four (4). This particle-type code of (4) designates an iron-rich particle, which can be assumed to consist primarily of iron oxide.

The data such as shown in TABLE 2 can be presented in a variety of forms. One of the most useful is to graph the particle size information. FIGURES 3, 4, & 5 are computer-plotted graphs of the equivalent circular diameter data for the five coal dust samples. Each figure is a number count plot of probability versus log of equivalent circular diameter (μm). In each case the data have been merged from an appropriate selection of magnifications. For example, the T-30, T-31, and T-36 plots result from size images recorded, two each, at 300 ×, 1000 × and 5000 × magnifications.

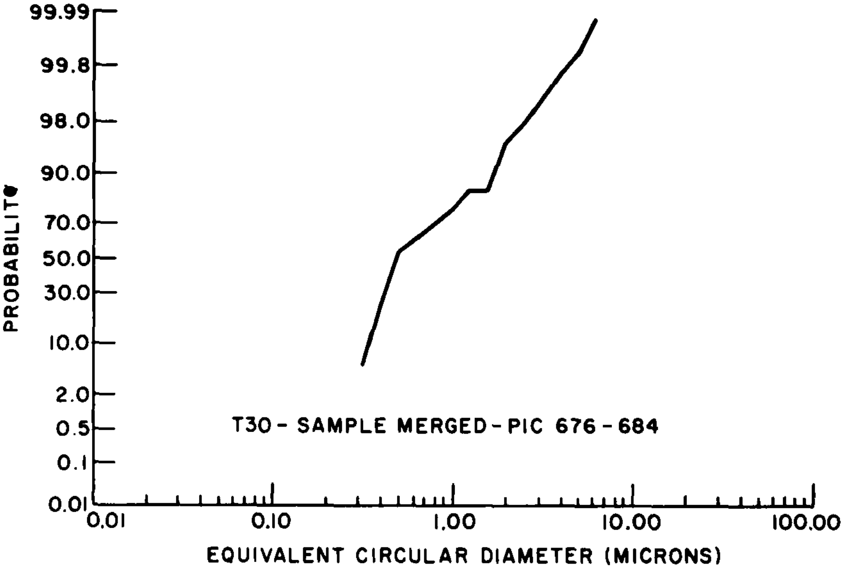

FIGURE 3. Probability versus log size for coal mine dust sample T-30.

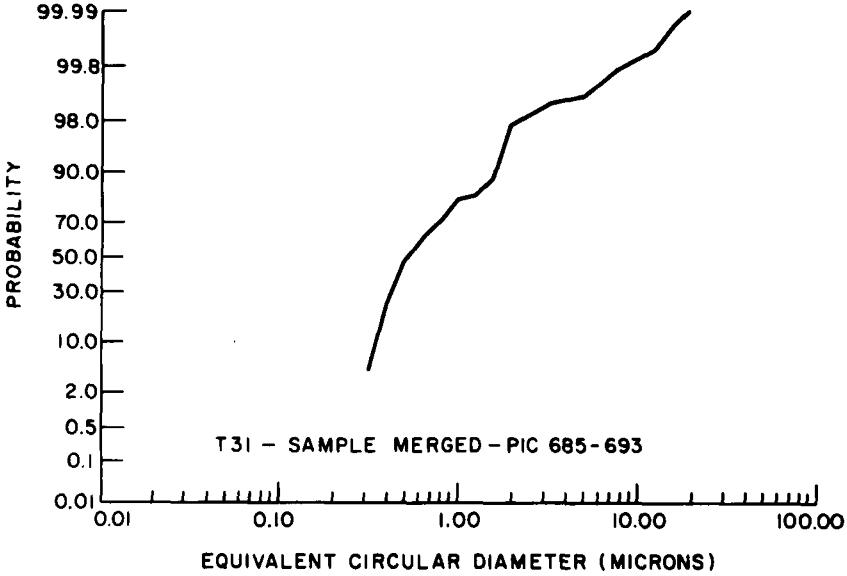

FIGURE 4. Probability versus log size for coal mine dust sample T-31.

An unexpected result for samples T-30, T-31, and T-36 was that less than one particle in 200 of size 0.5 μm and larger was a high-ash or mineral particle. From this result, it is inferred that most of the mineral and rock fragments in the dust are present as particles smaller than about 0.5 μm.

Under the conditions of these recordings, our x-ray sensitivity was not sufficient to identify positively the composition of particles in the size range of 0.1–0.5 μm. The instrumentation is currently being modified to increase sensitivity for the smaller size fraction. This modification involves changes in the beam control to increase the dwell time on small particles without unnecessarily prolonging the overall time for analysis.

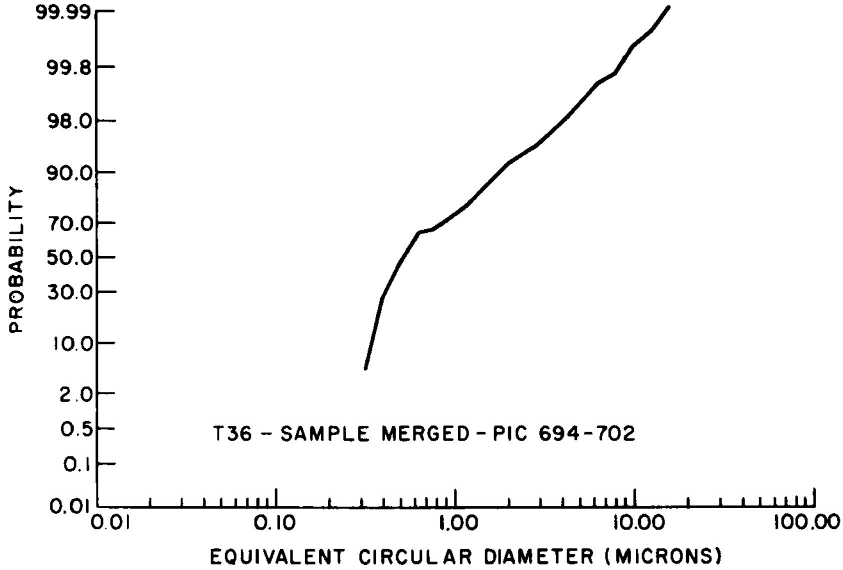

FIGURE 5. Probability versus log size for coal mine dust sample T-36.

ACKNOWLEDGMENTS

Pennsylvania State University staff working in the research program include: Professor G. G. Johnson, Jr., Professor H. A. McKinstry, Dr. H. Gorz, Mr. J. Lebiedzik, Miss K. Mayberry, and Mr. R. Hoover.

REFERENCES

1. WALKENHORST, W. 1970. New sampling methods for particulate matter with finely porous filters. Presented at the International Dust Conference, Bon-Bad Godesberg, June 9–11.
2. WALKENHORST, W. Results of a new method of determining the particle size distribution of suspended dust. Staub-Reinhalt Luft **30** (11).

3. DeNee, P. B. 1971. Mine dust characterization using the scanning electron microscope and dust samplers. Scanning Electron Microscopy (Part I). Proceedings of the Fourth Annual Scanning Electron Microscope Symposium, IITRI: 209–216. Chicago, Ill.
4. DeNee, P. B. Mine dust characterization using the scanning electron microscope. Submitted for publication in the AIHA Journal.
5. White, E. W., H. Gorz, G. G. Johnson, Jr. & R. E. McMillan. 1970. Particle size distribution of particulate aluminas from computer processed SEM images, Scanning Electron Microscopy. Proceedings of the Third Annual Scanning Electron Microscopy Symposium, IITRI: 57–64. Chicago, Ill.
6. White, E. W., K. Mayberry & G. G. Johnson, Jr. Computer analysis of multichannel SEM and x-ray images of fine particles. Pattern Recognition. In press.