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THE ELECTRON MICROSCOPE AND ITS ROLE IN DUST RESEARCH—PART III: OBSERVATIONS

By J. H. Talbot*

Summary

Observations of the shapes, electron optical densities, crystallinity and state of aggregation of particles have been made. Particles of drilling dust have been grouped into six classes according to these properties. Size distributions and number and area concentrations have been obtained for seventy-five samples. It is found that the Sichel distribution fits the observations at least as well as any other known distribution.† Diffraction and selected reflexion observations have revealed that the sub-microscopic particles of drilling dust consist mainly of certain salts occurring in mine water. The composition of sub-microscopic particles in other types of dust is being investigated but it is still too early to draw any conclusions.

Observations on the surface of finely ground quartz particles indicate that they may be free of the "disturbed layer" observed by other workers.

Particle sizing with the light microscope has been investigated with the electron microscope. This has revealed two systematic errors and a large random error in sizing. The smallest particle visible by conventional techniques is $0 \cdot 1 \mu$ but $0 \cdot 5 \mu$ is the lower limit of reliable observation. An improved light microscope technique has been devised resulting in substantial improvements in resolution and contrast and extending the range of observation well into the electron microscope region.

Introduction:

Most of the observations described were on airborne mine dust, but two special investigations of problems of vital importance in the study of dust and pneumoconiosis are also described. The term *dust* is here used to include all solid suspended matter in mine air. This is against a recommendation of the Electron Microscope Panel that the term

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†Statistical tests performed by Mr. A. D. Joffé, since this article was written, have shown that a two-term exponential distribution is at least as good a representation as the Sichel distribution.

dust be reserved for particles known to originate from the rock. Seldom, if ever, can one be certain that a particular particle has originated from the rock so that, following this recommendation, the term *dust* becomes redundant and one finds oneself using such confusing and ambiguous terms as *airborne scraping products.*

Qualitative Observations:

The observations described in this section I call qualitative because of the difficulty of obtaining accurate numerical estimates of the properties being studied. The principle observations of this type relate to shape, electron optical density, whether crystalline, and state of aggregation of the particles.

Particles of drilling dust have been grouped into six classes according to these observations.[1] Of these, only one is believed to be rock dust originating from the boring action of the drill. Another is believed to be carbon black arising from diesel exhaust smoke and from the introduction of atmospheric pollution from the surface with the intake air. The remaining four classes are believed to result from the atomization of drill feed water at the fronthead release ports. It is necessary to state that at the time these observations were made (from 1953 to early 1957) the short piston machine was almost universal. Now that this type of machine has been universally supplanted by the sealed spline machine it is to be expected that the conditions found will no longer apply. For this reason only those particles known to originate from the rock will be described. The remaining types have been described in detail before.[1]

Particles of rock dust have been studied mainly in experiments where some device has been used to exclude atomisation products and other extraneous airborne particles. Four methods have been used, viz. the suction hood invented by G. Hildick-Smith,[2] a wooden brattice, a collar box developed at the New Consolidated Gold Fields Research Bureau and a miniature aluminium brattice developed at the Dust and Ventilation Laboratory. Two of these methods, the suction hood and the collar box, were found to atomize the sludge so that some particles normally carried away by the water became airborne. Except for the greatly increased concentrations these particles were found to be similar both in appearance and in size distribution to those obtained by the other two methods.

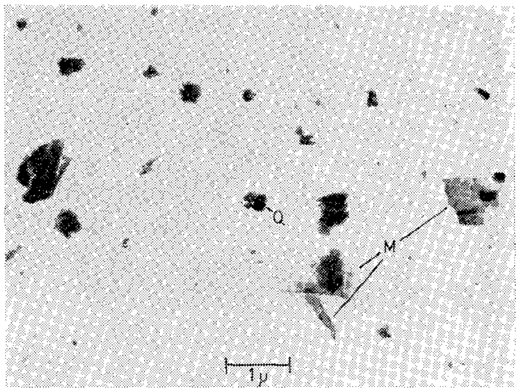

FIG. 1. *Sample of " hole only " drilling dust showing mica type and quartz type particles.*

Particles of hole only drilling dust in the microscope size range ($0{\cdot}2\mu$ to 5μ) have been sub-divided into two types viz. flake-like particles, established by electron diffraction to be mica, and particles having roughly equal extension in all directions, believed to be quartz. The two types are illustrated in Fig. 1. The experiments were done while drilling either into quartzite country rock or gold-bearing conglomerate. Under these conditions the mica particles were found to predominate. The sub-microscopic particles are characterized by sharply defined edges and high electron optical densities. Their composition is still unknown as the samples have not been sufficiently dense to be amenable to the electron diffraction method.

In samples of tipping dust the particles are similar to those of hole only drilling dust, except that the quartz type particles predominate in the larger sizes and the proportion of sub-microscopic particles is smaller. These observations apply equally to blasting dust where the larger particles are also found to be aggregated. Often particles of low electron optical density and indefinite outline are found in samples of tipping dust. These usually occur in clusters and are believed to be derived from water droplets.

Observations of particle shapes have recently been greatly aided by the develop-

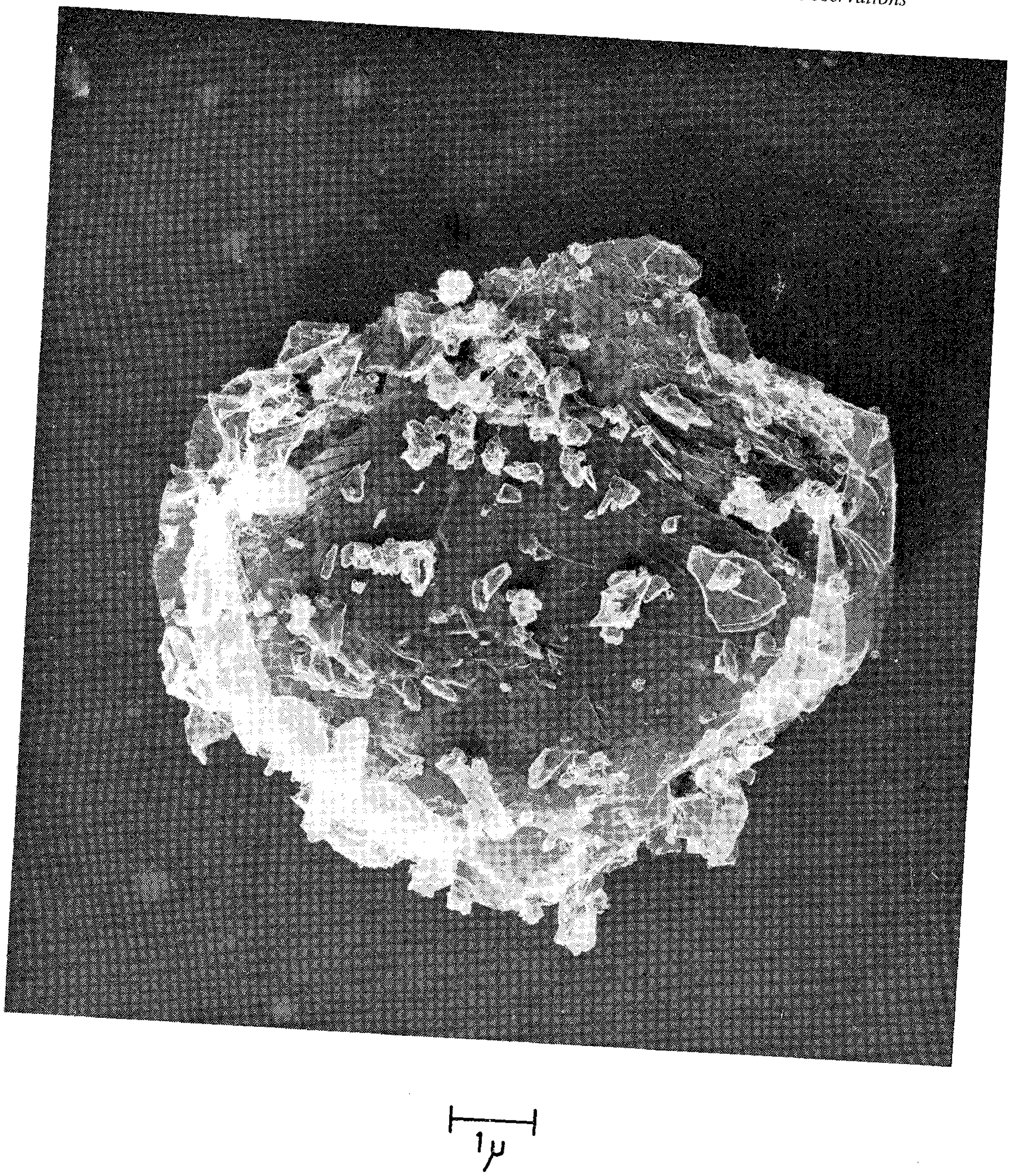

FIG. 2. *Surface replica of part of a sample of scraping dust. (Replica and micrograph by E. B. Kempis.)*

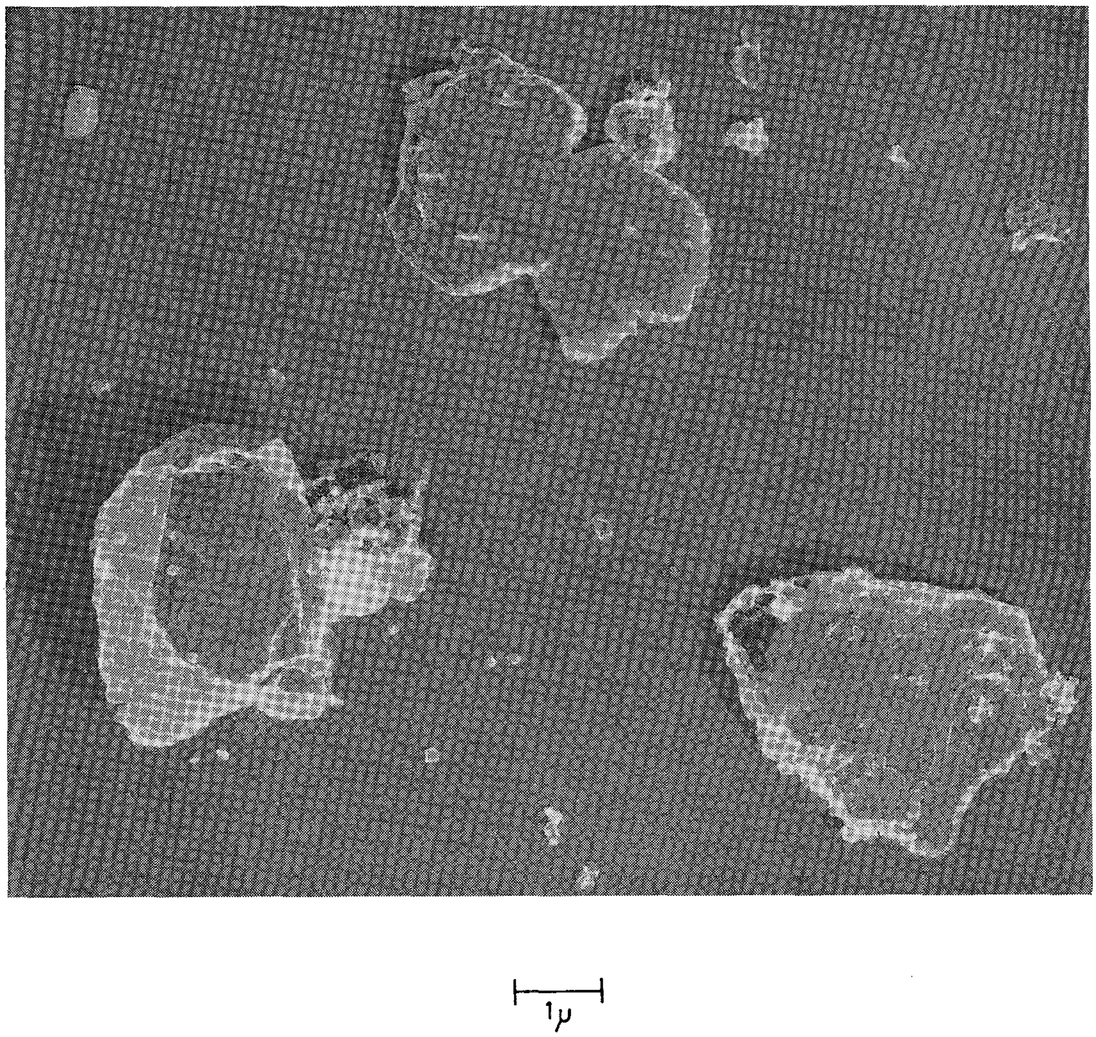

FIG. 3. *Surface replica of dust particles recovered from the lung of a deceased miner. (Replica and micrograph by E. B. Kempis.)*

ment of the surface replica methods[3] described in Part II. These have been used to compare the appearances of mine dust particles with those of particles found in the lungs of deceased miners. Examples of both types are given in Figs. 2 and 3, which are both good examples of this remarkable technique. Differences in appearance are thought to have been detected, but until such time as a more quantitative means of comparison is devised, our understanding of the differences will remain rather nebulous. This is one of the more intractable problems which remain to be solved.

The replica techniques have also revealed that what appear to be individual particles in ordinary micrographs are almost invariably aggregates.[4,5] This tendency to aggregation is often even more marked in specimens prepared by grinding bulk samples of quartz. Centrifuging and other well-known dispersing methods are largely ineffective in breaking up these aggregates.[5] This is a feature which can lead to serious errors in experiments to determine the biological effects of quartz powders; errors of 100 per cent. or more can result, in estimates of particle surface area, from this cause alone. In determining size distributions these aggregates are taken as individual particles. The size distributions are therefore dependent on the method of dispersion.

Size Distributions and Concentrations:

As mentioned in Part II, the principle object of dust studies with the electron microscope is to determine whether dust particles too small to be seen with the light microscope exist and, if so, whether they represent a health hazard. The quantity of dust is best expressed in terms of the number of particles of each size present in unit volume of air. Such an expression is called a size-frequency distribution.

To obtain size-distributions with the electron microscope, samples are taken with a thermal precipitator in which the cover glasses have been replaced by platinum specimen holders supporting silicon monoxide films as described in Part II. A series of electron micrographs covering an uninterrupted traverse across the dust strip is taken at a magnification of 2,500 ×. This normally requires twenty-six 3¼ in square plates. Measurement of the particles is effected by projecting an 8 × enlarged image of the photographic negative onto a screen, giving an overall magnification of 20,000 ×. Rigorous training of observers has resulted in size measurement of the images on the screen to at least as good accuracy as it is practicable to measure the magnification of the microscope in this type of work.

In addition to accurate size measurement of the sub-microscopic particles, the electron microscope has made possible the determination of the sizes of particles in the light microscope range with much greater precision. This is a result, not only of the greater resolving power, but also of the greater depth of field. The depth of field of the high power light microscope is so small that one can only focus on one section through the particle at a time. Parts of the particle above and below this section appear badly out of focus, giving a confused image from which accurate size determination is impossible.

The value of a size distribution may be greatly increased if a mathematical expression for it can be found. More accurate estimates of surface area and mass can then be made, and the possibility of extrapolating from observations over a limited size range can be considered. A number of theories of particle size reduction each leading to a different mathematical expression for the size distribution, have been proposed. Three of these have found favour with dust researchers. These are:—

(1) the exponential distribution, $y = Ce^{-\gamma x}$

(2) the power law distribution, $y = Dx^{-\delta}$

(3) the two-component Sichel distribution $y = Ae^{-\alpha\sqrt{x}} + Be^{-\beta\sqrt{x}}$,

where y = number frequency,
x = particle linear dimension, and A, B, C, D, α, β, γ, and δ are constants for particular samples.

In addition a two-component form of the exponential distribution is favoured by some authors for British coal mine dust.[6] Readers interested in the detailed characteristics of these distributions should consult references 6, 7, 8, 9, 10.

The size distributions of a total of about 80 samples of drilling, tipping and scraping dust have been determined.[11] With a few exceptions the Sichel distribution has been found to describe them quite accurately, and almost invariably it has proved superior to any other distribution in this respect.

Two typical observed size-frequency distributions are shown in Figs. 4 and 5. These distributions are plotted in a special way: *square root* of particle diameter against the logarithm of the frequency. The reason for plotting them this way is that if they conform to the Sichel distribution they will each appear as two straight line segments joined by a short arc, as indeed they do.

Distributions which conform to the power law appear as straight lines when the *logarithm* of the particle diameter is plotted against the logarithm of the frequency. A two-component power law distribution favoured by one author for South African

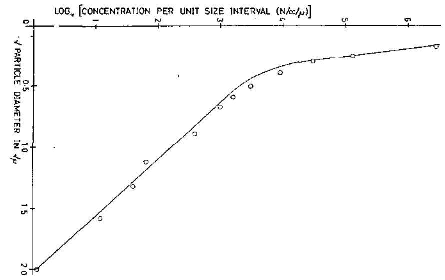

FIG. 4. *Size-frequency distribution of a sample of tipping dust.*

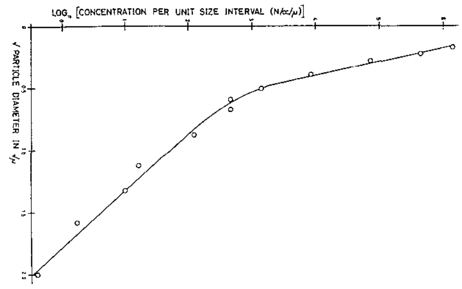

FIG. 5. *Size-frequency distribution of a sample of scraping dust.*

gold mine dusts[8] appears as two adjoining straight line segments. Now it so happens that the two distributions shown, when plotted in this manner, appear as moderately good pairs of straight lines. (The two distributions are not really as different as might at first appear to be the case.) This was regarded by proponents of the power law theory as support for that theory. A kink in the one straight line at a particle size of about 0·3μ was regarded by them as a sizing artefact.[12] The precision of the sizing techniques is such that we, who do the electron microscope work, are satisfied this is not the case. A recent investigation by A. D. Joffe (as yet unpublished) has revealed that just such a kink is to be expected if the distributions conform to the Sichel law. This is powerful evidence that the Sichel distribution is a more accurate description of our mine dusts than the power law. In fact the Sichel distribution may not have received the attention it deserves, especially overseas.

The principal numerical characteristics of the samples examined are summarized in Table I. In brief it may be said that, on the average, the sub-microscopic particles account for roughly 98 per cent. by number, 15 per cent. by area and 2 per cent. by mass, of all particles smaller than 5μ.

Composition:

As mentioned in Part II the electron diffraction method provides a sort of finger-print system whereby the crystalline constituents of the dust can be identified. Selected reflexion microscopy can then be used to give some indication of how these substances are distributed in the specimen. These methods were applied to samples of " total " drilling dust as early as 1954 and 1955. The sub-microscopic particles were found to be accounted for almost entirely by only two substances, calcium sulphate and sodium chloride.[1,13,14,15] The first is believed to originate from the practice of adding lime to neutralize the sulphuric acid in mine water; the second is perhaps the most common naturally occurring salt in mine water. Only a small number of samples, mostly from the Central Rand, were examined.

Application of the methods to other types of dust proved a much more difficult proposition and it was not until the beginning of the current year (1962) that this became possible. The high concentrations and high proportion of sub-microscopic particles in " total " drilling dust gave ideal specimens for the application of these techniques. The comparative coarseness and sparsity of other types of dust, such as that produced by tipping or scraping, made the application of these techniques difficult. Long before a sufficiently dense sample of the sub-microscopic particles could be obtained the field was obscured by the larger particles. The problem was eventually solved by first passing the air through a carefully designed air elutriator. The elutriator was designed to remove all particles with a Stokes' diameter larger than $\frac{1}{4}\mu$. In early laboratory and underground tests this design performance was achieved, but in the most recent sample some larger particles were found.

Only two samples of scraping dust have so far been examined. For satisfactory results about 25,000 c.c. of air has to be sampled. Since sampling is only carried out during operation of the scrapers and while there is no fear of contamination from other sources, it takes from two to five weeks to obtain one sample.

The small number of samples is partly offset by their large size, but it is still too early to draw any conclusions. The diffraction patterns contain very numerous spotty rings. In one case approximately 89 rings were measured — approximately 89, because possible displacement of spots by refraction or other causes made resolution of some rings uncertain. The results are of great value, however, in that the five other substances (quartz, mica, calcium sulphate, barium sulphate, sodium chloride) identified by X-ray diffraction as present in the larger particles, are found to be absent. Preliminary indications are, therefore, that most of the sub-microscopic particles do not come from the rock, but may have their origin in diesel exhaust smoke and atmospheric pollution from the surface.

TABLE I

Type of Dust	Surface area per unit volume ($\mu^2 cm^{-3}$)	Proportion of surface area accounted for by sub-microscopic particles
Drilling (mean of 2 samples)	1·9*	13%†
Tipping (mean of 46 samples)	840	17%
Tipping (return side only) ..	1,100	11%
Scraping (mean of 26 samples) ..	660	19%
Scraping (return side only) ..	750	17%

*Corrected to a dilution of 1,000 c.f.m.
†Subject to a very large statistical uncertainty.

Surface of Quartz Particles:

Ever since Briscoe *et al.*[16] discovered an anomalous effect in the solubility of finely ground quartz powders, the nature of the surface of quartz particles has been the subject of investigation, speculation and controversy. Kitto and Patterson,[17] who studied the solubility phenomena thoroughly, concluded that the effect could not be accounted for on the basis of particle size alone and attributed it to edge effects and the presence of a " Beilby layer," presumably of amorphous silica. In 1952 evidence in support of an amorphous layer came from X-ray diffraction observations.[18] This started a wave of intensive investigation when workers in many parts of the world, using a variety of methods, produced more and more evidence of this layer. One or two investigators disputed its existence.[19,20] One thing proponents of the theory could not agree on was the thickness of the layer. Estimates varied from less than a thousandth of a micron to several microns; the most popular was 0·03μ-0·06μ. Some workers, notably King and Nagelschmidt,[21] favoured a semi-crystalline, or *mosaic*, instead of an amorphous layer.

The nature of the surface of quartz particles is important as it affects modern theories of silicosis.

We became sceptical about the existence of this layer because of the enormous variations in the estimates of its thickness and because of the ease with which electron diffraction patterns could be obtained from the edges of quartz particles. Three types of observations were devised to test both the amorphous and mosaic layer theories.[22] Two of these had never been used before for this purpose and are worthy of mention. In the first, designed to test the amorphous layer theory, selected reflexion images of the edges of quartz particles,

(a) ground in acetone
(b) as (a) but subsequently etched in hydrofluoric acid to remove an outer layer from the particle, and
(c) as (b) but with a layer of amorphous silica about 0·03μ thick deposited over

it, were compared. The images of particles subjected to the treatment (c) were found to be markedly affected by the deposition of the silica. No such effect was observed in particles subjected to the treatment (a). It was concluded that if any amorphous layer existed it must be much thinner than 0·03μ.

In the second method carbon surface replicas of particles subjected to the treatments (a) and (b) were prepared by the method described in Part II, except that a shorter etching time was used. This resulted in incomplete removal of the quartz particles and many remnants could be found adhering to their replicas. Some were thin enough for transmission electron diffraction patterns of the types known as Kikuchi N and Kikuchi L to be obtained. Any mosaic structure of the type envisaged by King and Nagelschmidt would have been revealed by the patterns. No evidence of a mosaic structure was found.

A new theory was put forward to the effect that the observed phenomena could be accounted for by the presence of the numerous small particles which are always found adhering to the larger quartz particles, a phenomenon unknown at the time Kitto and Patterson published their paper. These small particles greatly increase the " edge effect." A team at the Max Planck Gesel-schaft in Germany came to the same conclusion independently.[23,24]

Subsequently a Japanese team, Sakabe *et al.*,[25] published results which leave little doubt that, in their experiments, a relatively thick amorphous layer was found on the particles. Their quartz was prepared by dry grinding with a mechanical agate mortar. It would appear that under these conditions an amorphous layer can be produced. A simple mechanism for its production has been suggested.[26] There was no evidence of the production of a *mosaic* sub-surface layer.

Particle Sizing with the Light Microscope:

Determination of size-frequency distributions of thermal precipitator samples with the light microscope forms an important part of nearly all dust research projects undertaken in this country. The techniques used are those described by D. G. Beadle.[27] The electron microscope provided, for the first time, a means of testing the accuracy of these techniques.

Particles in selected areas of dust samples were sized by experienced light microscopists. Maps were drawn showing the position, shape and estimated size of each particle. The selected areas were then photographed in the electron microscope and the particle sizes measured accurately from the electron micrographs. Before this could be done a method of transferring the sample from a cover glass to a supporting film had to be devised. The most satisfactory method was to deposit a layer of carbon over the specimen after the light microscope observations had been completed. The carbon film with the particles imbedded in it was then floated off the cover glass and mounted on a specimen carrier. As it was necessary to ensure an uninterrupted view of the whole selected area a special carrier was devised.

The following were revealed[28]:—

(1) Sizing of all particles is subject to a considerable random error, especially for sizes smaller than $0 \cdot 8\mu$.

(2) Particles larger than $0 \cdot 8\mu$ are, on the average, *undersized* in the ratio of approximately 4:5.

(3) Particles smaller than $0 \cdot 8\mu$ are, on the average, *oversized* in the ratio of approximately 5:4.

(4) Particles as small as $0 \cdot 1\mu$ can be seen.

(5) Below $0 \cdot 5\ \mu$ the observers started missing particles; approximately half the number of particles between $0 \cdot 1\mu$ and $0 \cdot 25\mu$ were missed.

It is felt that (1) and (2) can be improved by more rigorous training of microscopists. Little can be done about (3) except to accept the oversizing and allow for it. This also applies to (5) while conventional microscope techniques are used. However a new technique is described in the next section which promises to, at least partially, overcome some of these difficulties.

New Light Microscope Technique:

A technique has been devised which extends the range of observation of the light microscope well into the region which was formerly the preserve of the electron microscope. While some spectacular results have already been achieved, it must be emphasized that the technique is still very much in the experimental stage.

In Part I it was mentioned that, in passing through the specimen, the light rays undergo certain changes. Basically, one or more of three things may happen: modulation of the amplitude, frequency, or phase may occur. A particle which produces amplitude modulation reduces the intensity of the light and appears darker than its surroundings. A particle which produces frequency modulation will differ in colour from its surroundings. Phase modulation cannot normally be detected by the eye, so that a particle producing only phase modulation is normally invisible. The phase contrast microscope converts phase modulation to either amplitude or frequency modulation, making phase modulating objects visible.

Quartz particles produce phase modulation only. This is why they appear transparent and colourless. The phase modulation causes some of the light rays to change direction. This is known as scattering or diffraction. It is the function of the lenses to collect these rays and focus them back into the part of the image corresponding to the part of the particle from which they originated. If the lenses could collect all the scattered light and focus it perfectly, there would be no contrast and no image. But since the lenses cannot do this, some small contrast arises. We see the particles, therefore, only because of the imperfection of the microscope. The phase contrast microscope has proved unsuccessful with very small particles, probably on account of the small magnitude of the phase modulation.

The new technique greatly increases the visibility of small quartz or dust particles by

(a) converting them to amplitude modulators, and
(b) by increasing their size by a known amount.

This is achieved by evaporating aluminium onto the sample in a high vacuum at an angle of 3° to the plane of the cover glass. During the evaporation the cover glass rotates in its own plane about an axis through its centre. Edges of the particles are thus coated with aluminium leaving the cover glass almost perfectly transparent. Tests were made with 0·088μ diameter polystyrene spheres, which are normally completely invisible in the microscope. A coating of approximately 0·03μ of aluminium, increasing their diameter parallel to the plane of the cover glass to approximately 0·148μ, made them so distinct that not even the most careless microscopist could miss any of them.

Another advantage is that oil immersion does not affect the visibility of particles coated in this way so that full advantage can be taken of both the objective and illuminating apertures. The result is a gain in resolution by a factor of almost 2 over present techniques.

Conclusion:

Nine years ago, when this work was commenced, it was not known whether any sub-microscopic particles were present in mine air. Today we not only know that such particles are present, but also the quantities in which they are present. We are learning what they are composed of, and when this project is completed we shall endeavour to discover to what extent they are harmful.

The fact that they conform fairly accurately to the Sichel distribution means that we can extrapolate from observations over a limited size range. However, to extrapolate over the full range of interest (roughly 0·01μ to 10μ) it is necessary that the observations be taken over a range which makes the determination of both the α and β constants possible. With the present light microscope techniques the determination of the β constant is often unreliable. However, a new technique which may overcome this has been devised. Extrapolating is a safe procedure in that any error will lead to an over-estimate of the sub-microscopic particles. This is because invariably any deviation from the Sichel law has represented a deficiency of the smallest particles in the observed distributions.

Since quartz and dust particles show varying degrees of aggregation, even when dispersed by the most efficient means known, and since the condition of the surface of quartz particles appears to depend on the previous treatment, these factors may have to be taken into account in biological experiments and in assessing the dust hazard.

Acknowledgements:

Much of the work described was performed by my co-workers mentioned in Part II. I would like to express my appreciation to the numerous mining companies which provided sites for experiments and for sampling, to Mr. P. H. Kitto for helpful advice and suggestions, to Messrs. J. S. Maritz, H. D. Miller and A. D. Joffe for their statistical work and to the staff of the Dust and Ventilation Division for taking the samples.

But for the initiative of Mr. D. G. Beadle in persuading the Chamber to invest in electron microscopy it is unlikely that any of this work would have taken place.

Finally, I would like to thank the Transvaal and Orange Free State Chamber of Mines for permission to publish this article.

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SUGGESTED ANSWERS TO VENTILATION QUESTIONS IN THE MINE MANAGERS' EXAMINATION

Editor's Note

It is apparently common knowledge that the questions on ventilation—and particularly those involving ventilation calculations—prove one of the major difficulties facing candidates for the Mine Manager's Certificate of Competency in South Africa.

In an endeavour to provide these candidates with some assistance, the Society has decided to publish from time to time in the Journal, a number of suggested answers to problems which have recently been set in this examination. It is hoped that readers will draw the attention of candidates to these questions and answers. One set of answers appears on opposite page and further selections will appear in later Journals.