With the Calibrator mounted on this base, it is believed that tests can be conducted at all times and that resolution testing even for research purposes (if such need should arise) should be practically unlimited, even using high resolution spectroscopic plates.

A specially built air-conditioned room, with temperature controlled from that room, will house all the calibration equipment. Cameras and cones coming into the room for testing will be brought in a full day prior to testing or calibrating. The setting, pinning and doweling of the fiducials will all be done in the same temperature controlled section.

Although this equipment was built for production work Fairchild believes that the quality is such that if at any time the need arises, it can be used as a reliable research tool.

The success which we are now in position to predict for the Fairchild Calibration project will be largely due to collaboration between men prominent in the field of photogrammetry and the Fairchild personnel. There are many who have contributed generously of their time and knowledge, but in particular the authors would like to express their appreciation of the constant interest and encouragement in addition to substantial aid offered by Drs. *Irvine C. Gardner* and *Francis E. Washer* of the National Bureau of Standards and Dr. *L. E. Howlett* of the Canadian Research Council.

At Fairchilds, Mr. *Irving Doyle*, Engineer in Charge, has guided the project, making available his many long years of experience in the field of aerial cameras.

THE STUDY OF SIZE AND SHAPE BY MEANS OF STEREOSCOPIC ELECTRON MICROGRAPHY*

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IT IS with some trepidation that a microscopist presumes to address a Society such as yours which is concerned basically with the *measurement* of photographic records; he realizes, and a very brief perusal of a few articles in the current magazines quickly confirms, the fact that a microscopist usually is concerned with qualitative aspects of the pictures he takes. True, he wants to know as accurately as possible the magnification or scale of his micrograph, and there are a few cases such as measurement of particle size histograms where detailed measurements are the principal purpose. But in the great majority of cases, he is interested in observing characteristic features of the material depicted in the micrographs. For example, a biologist observes in electron micrographs that certain fibers in connective tissue exhibit a periodic substructure; or a metallurgist observes in optical or electron micrographs certain tree-like markings which he calls dendrites. These observations are analogous to and may be compared with such common observations as that a freshly plowed field exhibits furrows, or that a river system consists of a river with large numbers of branching tributaries. Usually, what the microscopist would like to find out is the cause of these characteristic features, and he is only secondarily concerned with detailed measurement.

* Paper read at Semi-Annual Meeting of the Society, Institute of Geographical Exploration, Cambridge, Mass., September 21 and 22, 1950.

1. MICROSCOPE RESOLUTION, MAGNIFYING POWER, AND DEPTH OF FIELD

The electron microscope is entirely analogous to the optical one. In both cases we have the thing we want to see, called the object. An objective lens is used to form a real or virtual image of this object, which is then projected by a projector lens on a photographic plate. With visible light, the image can of course be viewed directly by using an eyepiece lens instead of a projector lens, but with ultraviolet light or electrons it must be projected on a fluorescent screen

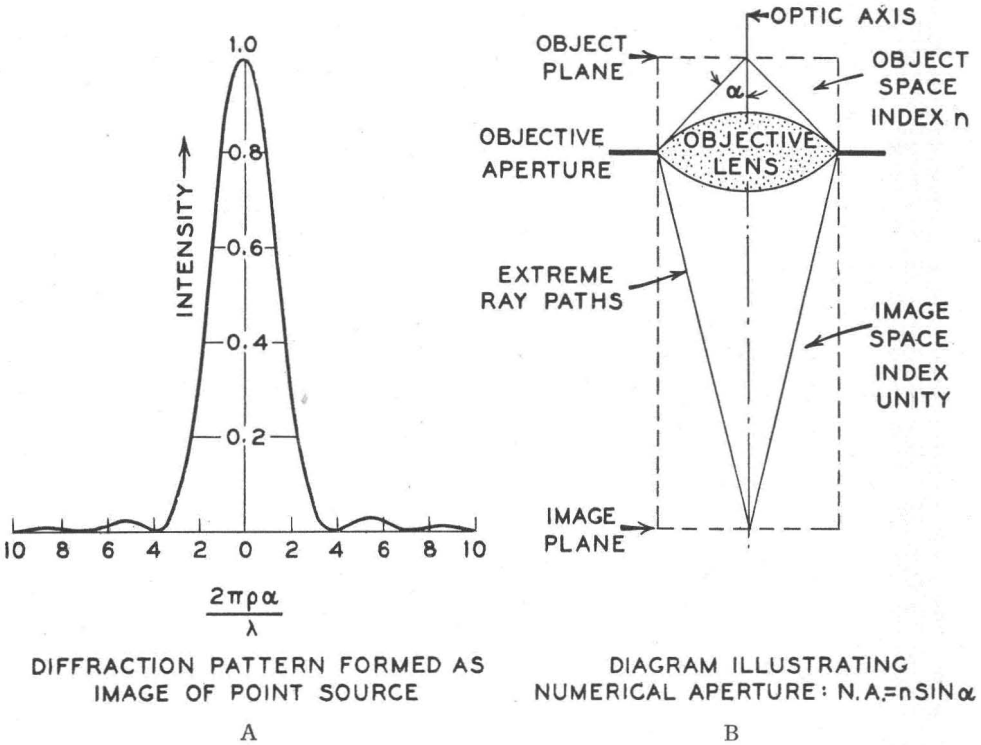

FIG. 1 (a). Intensity distribution in the image plane when a point source is imaged by a lens of aperture angle α. (b). Diagram illustrating numerical aperture.

screen for visual study. The electron lenses in the electron microscope consist of short distances along the axis of the microscope within which intense magnetic or electric fields exist.

In both cases the possible magnification depends on the resolution. The human eye can see as separated, two luminous points when they subtend an angle of about one minute. If the points are at a distance of 10 inches, which is about as near as most eyes will focus, the points can be seen as separate if they are more than about a tenth of a millimeter apart. This distance is called the resolution of the unaided eye. The basic reason for the existence of this limit is that the pupil of the eye is an aperture restricting the cone of light received from the object; diffraction due to this aperture causes the area on the retina illuminated by a point source to be not a point but a central bright disk surrounded by concentric dark and light circles. The radii of the central disk and of the concentric circles are inversely proportionate to the diameter of the pupil. This is illustrated in Figure 1, in which the ordinate is intensity and the ab-

scissa is the radius ρ measured from the geometrical image point, multiplied by $2\pi\alpha/\lambda$ to make the curve general and applicable to all values of aperture angle α and wavelength λ. Two adjacent point sources appear as separated only when the central disks are in effect separated. The minimum distance apart of two observably distinct sources is called the resolution. Theoretically this depends on the forms of the sources, i.e., whether they are lines, points, or some other form, and on the conditions of illumination, but it is not less than the radius of the first minimum in Figure 1. Setting the resolution R equal to this value of ρ yields the equation $R=0.61\ \lambda/n\ sin\ \alpha$. On the basis of this formula the unaided human eye should be capable of resolving two points separated by 0.03 mm, but lens defects of the eye result in the higher value of 0.1 mm.

INSTRUMENT	LEAST DISTANCE WHICH CAN BE RESOLVED	MAXIMUM NORMAL MAGNIFYING POWER	MAXIMUM USEFUL MAGNIFICATION	OBJECTS RECOGNIZABLE AT THE MAXIMUM MAGNIFICATION
UNAIDED HUMAN EYE	10^{-2} CM			.004" WIRE
MICROSCOPE. VISIBLE LIGHT	1.8×10^{-5} CM	550	1650	BACTERIA
MICROSCOPE. ULTRA VIOLET LIGHT	10^{-5} CM	1000	3000	LARGE VIRUS PARTICLES
ELECTRON MICROSCOPE	10^{-7} CM	100,000	300,000	LARGE MOLECULES

FIG. 2. Comparison of light and electron microscopes with respect to resolution, normal magnifying power, useful magnification and objects resolved.

The optical microscope produces better resolution, primarily by a large increase in α, which has an obvious relation to the F-numbers of camera lenses. But as α is increased beyond about 45°, which corresponds to $F=1$, the law of diminishing returns operates sharply. For further improvement in resolution, the wave-length λ/n may be decreased by decreasing λ or increasing n. Figure 2 summarizes the practical limits of present microscopes. Normal magnifying power is the ratio of the resolution of the unaided eye to that of the microscope as calculated by the above equation. Magnification in excess of this is sometimes called empty magnification. However, because many eyes cannot resolve 0.1 mm., and further because it is a strain on the eye to be constantly trying to resolve the finer details, it is customary to print micrographs at magnifications two or three times the normal magnifying power. This higher magnification is called the useful magnification.

The figure shows resolution and magnifying power for the electron as well as the optical microscope. The reason why there is such a large step is that the wave-length is only about one one-hundred-thousandth that of visible light. A more detailed comparison of electron and optical microscopes is shown in Figure 3. The wave-length of a beam of electrons is fixed by their energy and may be varied by varying the voltage. The electron microscope operates at a very small aperture, about F-150 in terms of F-numbers. Because of the extremely short wave-length, the resolution is adequate even at this small aperture to resolve down to large molecules. Further improvement in resolution would be of doubtful utility because detail on the scale of the resolution seldom gives rise to sufficient contrast to be detectable.

The optical microscope, when operated to obtain the best possible resolution, has F-number less than unity and hence very poor depth of focus. Even though

the electron microscope operates at about 50 times the normal magnifying power of the optical microscope, it still has depth of focus 20 times greater, on account of its very small aperture (large F-number). This great depth of focus makes it possible to take stereoscopic pairs of micrographs, even at the highest magnifications. In stereoscopic vision, each eye of the observer must view the same field from slightly different angles. With the electron microscope, this effect is achieved by tilting the specimen, first to one side, taking one picture, then to the other side and taking the second. If this technique be used with the optical microscope, the limited depth of field at the higher magnifications reduces the area of field in good focus to extremely small values. Although this limitation can be removed either by displacing the object laterally or by using one half

PROPERTY	ELECTRON MICROSCOPE (40-100 KV ELECTRONS)	LIGHT MICROSCOPE
WAVELENGTH λ	0.07 - 0.04 Å	6500-2500 Å
RESOLUTION LIMIT (THEORETICAL)	14 - 6 Å (NA = 0.003)	2800-1100 Å (NA = 1.4)
RESOLUTION (PPACTICAL)	20 - 50 Å	2800-1100 Å (NA = 1.4)
NORMAL MAGNIFYING POWER (PRACTICAL)	~ 30,000	360-900 (NA = 1.4)
DEPTH OF FIELD AT NORMAL MAGNIFYING POWER	~ 20,000 Å (NA = 0.003)	2000-800 Å

FIG. 3. Important properties of light and electron microscopes.

of the objective lens to form one image, the other half to form the other, the depth of focus at the highest magnifying powers is less than the resolution and hence insufficient to portray vertical relief. Since depth of focus is inversely proportional to the square of the normal magnifying power, whereas the linear dimensions of the image field are of course proportional to the first power, these limitations decrease as magnification is reduced and are usually unimportant when the magnification is less than 100.

2. STUDY OF SURFACES

The next series of figures (Figures 4–10) show stereoscopic pairs of electron micrographs of the natural surfaces developed during sintering of thin flakes of a nickel-manganese oxide. Sintering may be defined as the growing together or welding of particles at temperatures below the meling point. It occurs at appreciable rates when the temperature exceeds about 0.6 T_m, where T_m is the melting point in absolute temperature (°K). The flake was originally prepared by mixing two very finely pulverized powders of the oxide with a plastic binder. The average particle size of one of the powders was about 2 microns, the other about 0.01 micron. The mixture was spread on a glass plate and allowed to dry, after which the layer was peeled from the glass and cut into 5 mm. square pieces.

Figure 4 shows the surface produced by prolonged sintering in air at a temperature (1,030°C.) much below the melting point (about 1,500°C.). Recrystallization has resulted in production of grains much larger than the

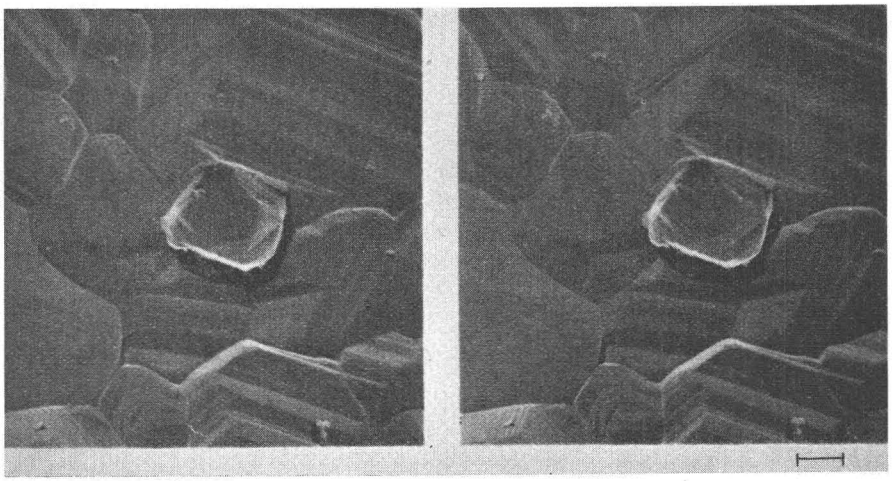

FIG. 4. Natural surface of a nickel-managanese oxide flake developed by heating 64 hours at 1,030°C. The small particles are crystals of MgO deposited from MgO smoke for the purpose of determining stereoscopic up-and-down. Large grains with well-developed crystallographic surfaces. Compact structure with no inter-granular pores. The marker shows the length of a micron.

original large particles. These grains are closely packed, with no observable intergranular cracks or pores, indicating a very compacted structure. Figure 5 shows the surface developed by heating to 720°C. for a few minutes. This treatment vaporizes the plastic binder and leaves the larger particles embedded in a matrix of the small ones. Figures 7, 8, and 9 show progressive stages of the sintering when the flakes are heated respectively to 800°C., 900°C., 1,000°C., for a few minutes. Up to 1,000°C. the original large particles may be distinguished as a group, from the group of growing small particles. Of course, the larger particles also grow within this range of firing temperature by absorption of some of the small ones, since Figures 7 and 8 show examples of absorption of small particles into large ones. But for reasons which will become clear later, it

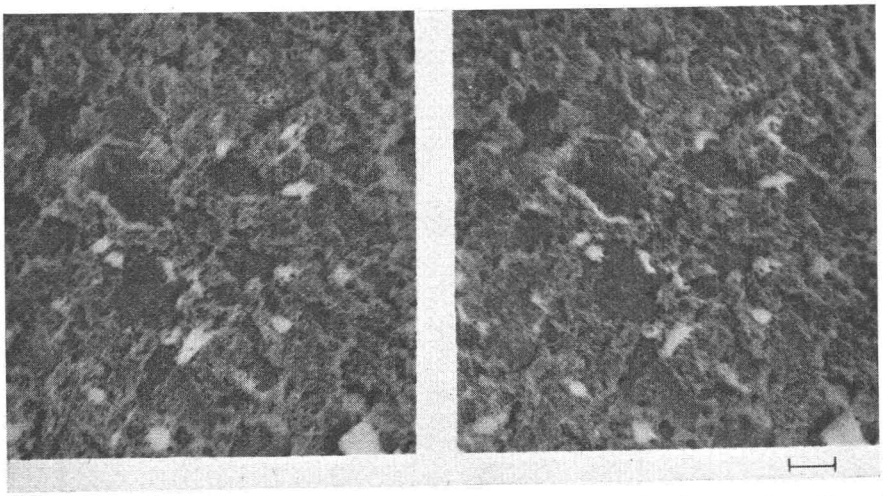

FIG. 5. Ni-Mn-O flake surface after heating briefly to 720°C. The large and small particles of which the flake was originally molded are evident, with the large embedded in a matrix of the small particles.

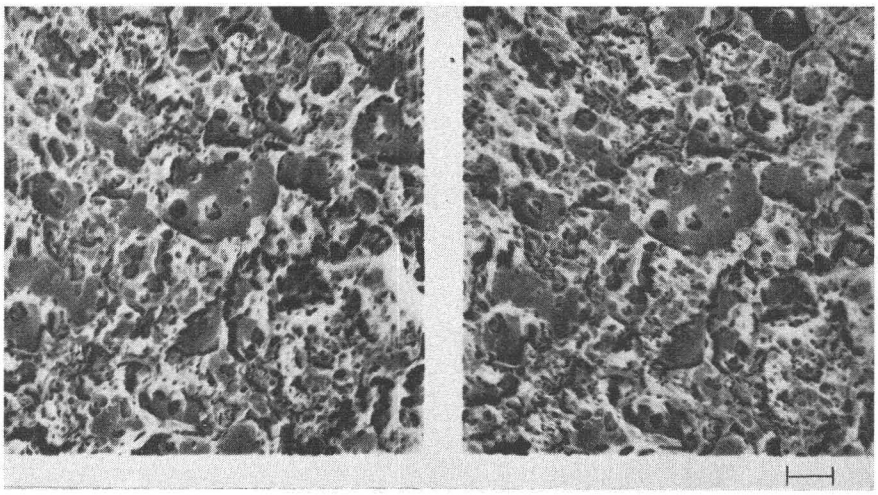

FIG. 6. Ni-Mn-O surface after heating briefly to 800°C. The small particles originally touching the larger ones have been absorbed. The particles between the large grains have sintered to form somewhat larger particles. Some of these may be seen in the process of absorption on the surfaces of the large particles.

is not possible to examine the same area following successive firings. Accordingly the amount of this growth can only be measured statistically, and is found to be quite small relative to the size of the large particles. The reason for this is that the layer of small particles immediately surrounding a large particle is absorbed rapidly by the large particle, leaving an annular gap, and it is only later and at a higher temperature that sintering occurs between the particle and its growing smaller neighbors. All this behavior can be deduced from *stereoscopic* study of the original micrographs, although it may not be evident in the illustrations because of the limitations of the half-tone method of reproduction. It cannot be deduced by even the most careful examination of single micrographs.

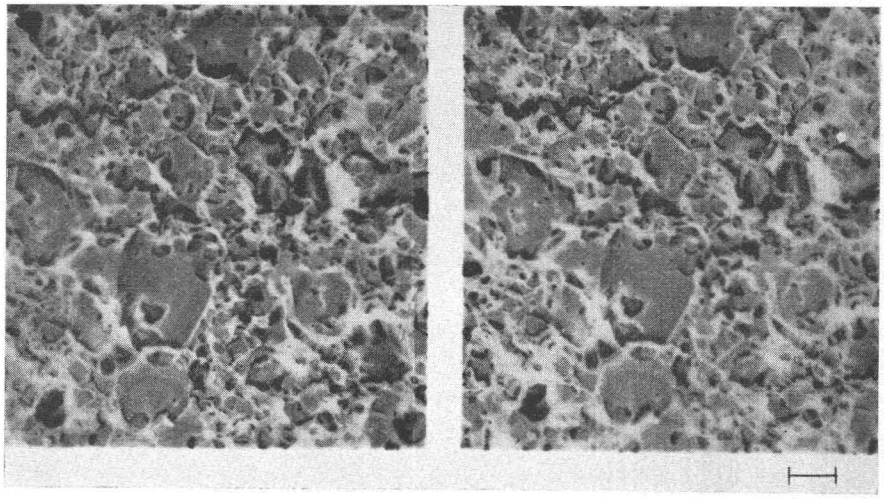

FIG. 7. Ni-Mn-O flake surface after heating briefly to 900°C. The processes described under Figure 5 have proceeded further.

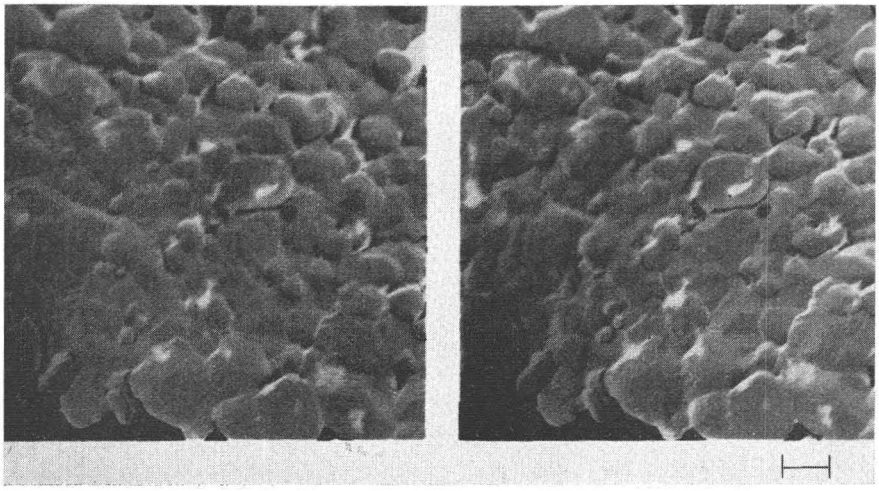

FIG. 8. Ni-Mn-O flake surface after heating briefly to 1,000°C. The larger particles are barely identifiable, the sintering of the smaller particles having now produced many particles in the size range of the original large particles. Beginning of development of crystallographic planes, which indicates that particles are now individual crystallites, Inter-granular pores are numerous but small.

Stereoscopic electron micrographic examination of surface structures, utilizing both the high resolution and great depth of focus of the electron microscope, usually reveals far more about the surface, and this additional information as compared to single micrographs is often the key to the interpretation of the structure.

This is illustrated in figure 9, which shows the surface of a flake which has been heated for 120 hrs. at 950°C. Examination of either picture separately shows merely a confused pattern of light and shade, grains being distinguishable in some areas, but apparently partially covered with "shrouds" that obscure the grains in other areas. The stereoscopic pair is mounted with the right-left orientation such that a view is obtained from *inside* the flake (a worm's-eye

FIG. 9. Ni-Mn-O flake surface after heating 120 hours at 950°C. Mounted so as to give a view from inside the surface. "Shrouds" indicate presence of large inter-granular pores, relatively few in number.

rather than a birds-eye view!). The shrouds are immediately seen to be areas of the grains lying more or less under the tops of the grains—in other words, to be due to cracks or pores between grains. Thus stereoscopic observation permits the deduction that prolonged sintering at temperatures below 1,000°C. results in a structure characterized by large pores. This is a culmination of the process mentioned above, that large particles rapidly absorb contiguous small particles, forming annular rings.

If, however, the flake is rapidly heated to a temperature in excess of 1000°C., and held there a long time, Figure 4 has shown that a very compact structure is obtained. This is explained by the fact that at high sintering rates, the mass of small particles sinters more uniformly to form intermediate size particles (Figure 8), separated from each other by pores small compared to those of

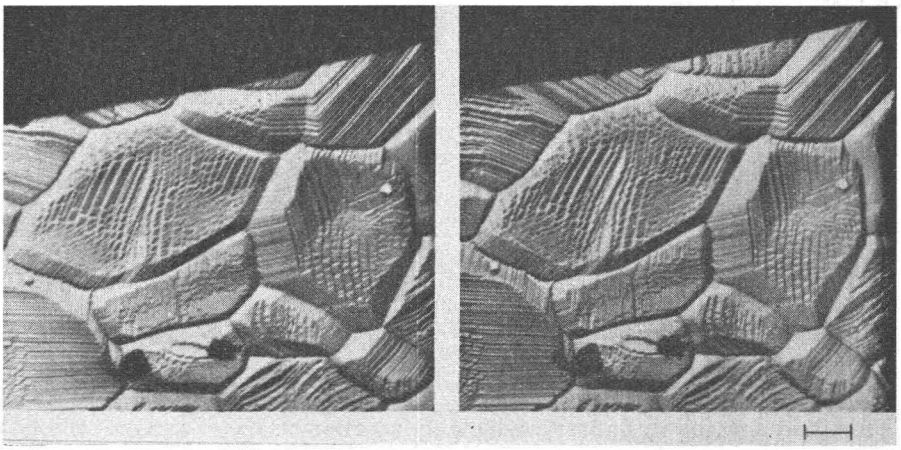

FIG. 10. Ni-Mn-O flake surface after heating briefly to 1,250°C. Large crystallites with well developed and variegated crystal surface planes.

Figure 9. During prolonged heating, sintering can now progress at the numerous contact areas, resulting in reduction of pores and a compact structure.* A compact structure can also be obtained by rapid heating to a more elevated temperature, holding this temperature for a short time, then allowing the flake to cool. Figure 10 shows a surface area of a flake so treated, and it is seen to be quite compact, but with a more variegated crystallographic surface pattern. In the use which is made of these flakes as thermistor elements, absence of pores, which contribute to the electrical noise, is essential.

Although in the case of grains such as those depicted in Figures 4, 8, and 10, single micrographs are adequate for measurement of grain size, this is not true for Figures 5, 6, and perhaps 7, where stereoscopic relief is essential to determine the location of the smaller particle boundaries. Figure 11 shows a plot of particle size as a function of firing temperature, the curves showing the ranges of the two groups of particles. These merge at a firing temperature of about 1,000°C., and at higher temperatures show characteristic crystallographic surface markings indicating that each particle has become an individual crystallite. The curves apply to a standard firing cycle in which the temperature is raised

* The original particles were in a highly strained state as a result of pulverization, and the fact that this strain is relieved rapidly at temperatures above 1,000°C. is an important contributing cause.

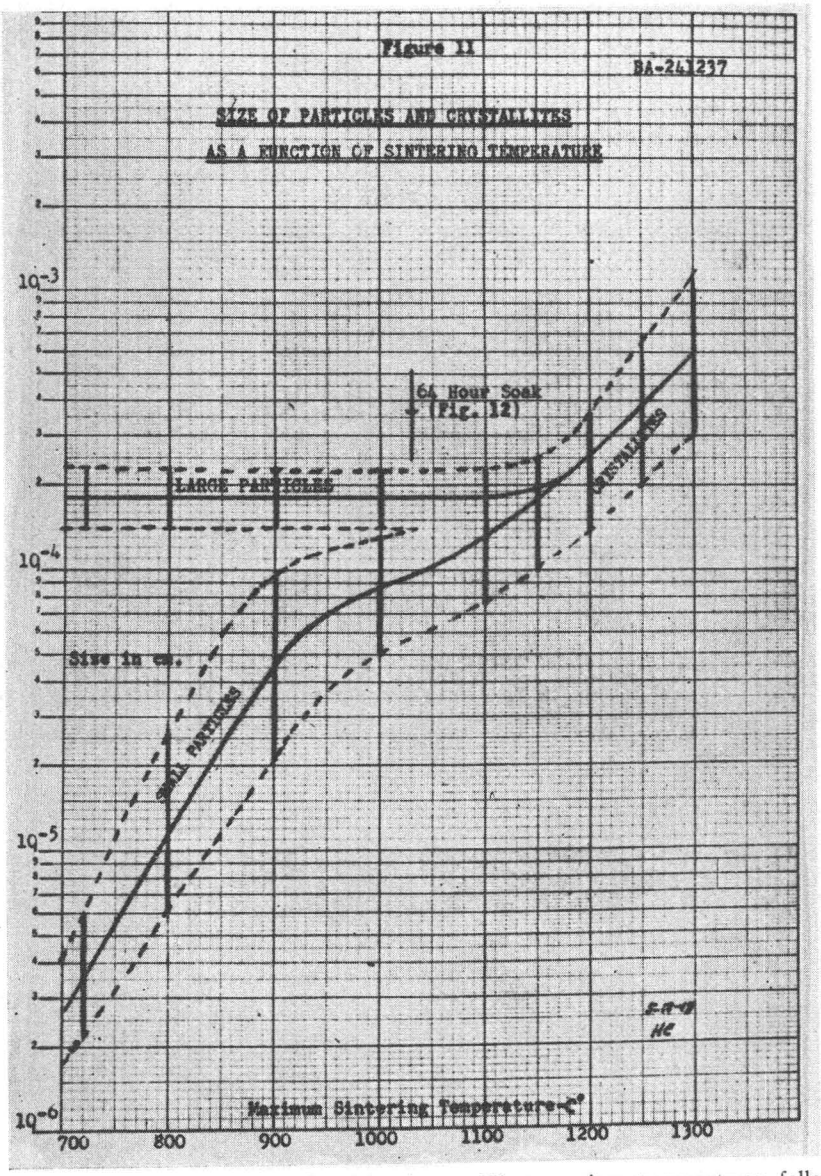

FIG. 11. The effect on particle size of heating rapidly to various temperatures, followed by rapid cooling. The group of 2 μ particles does not change up to 1,000°C., but the group of 0.01 μ particles sinters to form progressively larger particles, merging with the first group at about 1,000°C., above which the particles are individual crystallites with well developed crystallographic surfaces. Prolonged heating at 1,030°C. has about the same effect as rapid heating to, and cooling from, 1,250°C.

rapidly to the indicated value, then the furnace power is cut and the furnace allowed to cool.

By employing the methods of quantitative stereoscopy on electron micrograms, profiles along selected lines may be obtained. Figure 12 shows such a profile along a particular line in a portion of Figure 10. It shows that the general surface level varies by about a micron, and that the depth of grain boundary depressions ranges up to about 0.2μ. Since the flake is only about 10μ thick,

these are significant observations. The vertical resolution of such elevation difference determinations is not all that could be desired. A stereoscopic angle of 8° was used in making the pairs. For reasons discussed below, the horizontal resolution is perhaps 0.01μ, and hence the vertical resolution is only about 0.07μ. This situation could of course be improved by using a larger stereoscopic angle, but for various reasons this process is limited. Angles as large as 20° have been used in electron microscopy, in which case the vertical resolution is about three times as large as the horizontal. It may be mentioned that in ex-

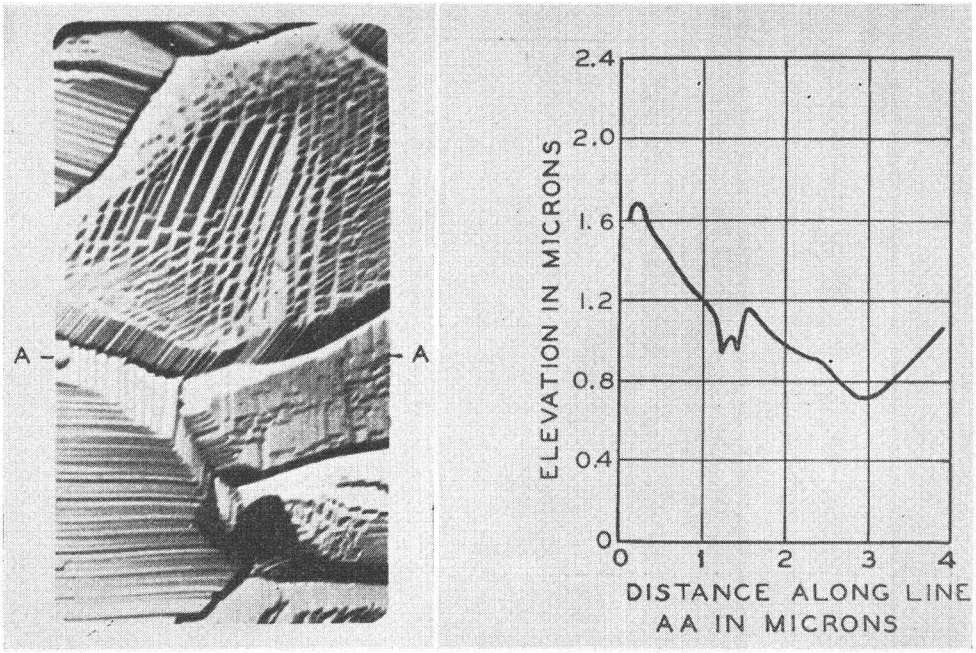

FIG. 12. A portion of the micrograph of Fig. 10, with a profile along AA. Surface level varies about 1 μ and grain boundary depressions are about 0.2 μ.

amining stereoscopic electron micrographs, one is often aware of elevation differences somewhat less than the calculated vertical resolution, but attempts to measure them quantitatively fail. The awareness of a small elevation difference is probably due to perspective caused by shading effects, and is particularly evident when the shading has considerable symmetry, as in the crystallographic surfaces of Figures 4 and 10.

One does not, in the electron microscope, observe the actual surface. All electron microscopes are transmission-type microscopes, that is to say, the electrons must pass through the object actually mounted in the microscope. But 50 kv electrons cannot pass through objects thicker than a few thousand Angstroms (i.e., thicker than about the wavelength of visible light). So recourse is made to thin-film replicas of the surface of interest. A detailed description of the many methods of replication will not be attempted. The simplest one is to wet the surface with a dilute solution of a plastic such as collodion, then allow the solution to dry, and peel the thin plastic film from the surface. This yields a plastic film of locally varying thickness, the local variations being related to the surface topography in a not-very-simple manner. These thin plastic films

are unsatisfactory because they are somewhat unstable under the intense electron bombardment they undergo when in the electron microscope. Moreover, because of the low density (about unity) of the collodion, they must be made 500–1,000*A* thick in order to produce a fair range of contrast. This limits their ability to resolve detail. However, if such films of a thickness of 100–200*A* are mounted in an evacuated bell-jar, and a layer of metal deposited on the surface which had been in contact with the original object surface, by evaporation of the metal from a suitable source, their stability is much improved. Further, if the metal is deposited at an angle of incidence far from the surface normal, it will be of varying thickness dependent on the local topography. Such a replica

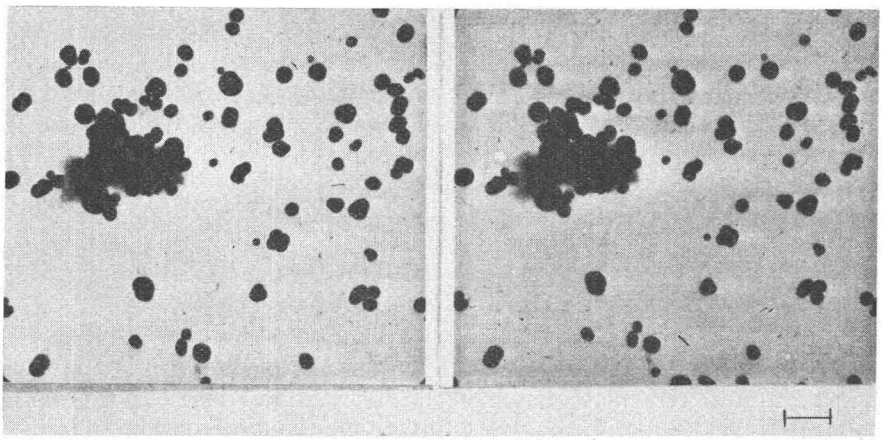

FIG. 13. Carbon black particles about 0.25 microns in diameter. The cluster is seen stereoscopically to be piled up vertically. Reticulation, i.e., the physical joining of particles by short, narrow necks, is probably the reason for grouping together of some of the particles.

is called a shadow-cast plastic replica, because the major surface features actually cast "shadows" in the metal film. It is considerably used, particularly in metallurgical studies.

A second method of replication consists of molding the original object in a plastic block (usually polystyrene) at suitable temperature and pressure. After cooling, the plastic is cut away by sawing and filing until the object is freed from the plastic, except at the surface which it is desired to study. A sharp mechanical blow then serves to separate these two surfaces, and the surface of the plastic block is then a mold of the desired surface.* This block is mounted in the evacuated bell-jar and a layer of silica of proper thickness deposited on the mold by evaporation from a suitable source (See Figure 15). (Several other materials besides silica are also useful for this purpose, the most common one being silicon monoxide.) The surface is then scored into small squares, and the block is then placed in a suitable solvent for the plastic. After a time the small squares of silica film drift loose from the block and are picked up by means of pieces of fine mesh screen. They are rinsed to remove the last traces of plastic. Silica films of this type are very stable in the electron microscope, and preserve to a high degree the topographic form of the surface, even to the extent of reproducing reentrancies of the surface, as was illustrated in Figure 9. Because

* In the case of the thin ceramic flakes depicted in Figs. 4–10, separation was accomplished by dissolving the flake in HCl. This explains why observations on replicas made by successive firings of a single flake were not possible.

of this dimensional stability, many electron microscopists prefer them to the shadow-cast plastic replicas previously described.

It may be shown that the resolution of a replica film tends to be about half its thickness. If an electron beam of 50 kv energy is used in the microscope, impinging on a photographic plate to produce blackening, a barely perceptible difference in blackening is produced when a film of *mass* thickness about 0.7 μg./cm^2 is placed in its path. In order to have many shades of contrast available in a micrograph, the average film thickness must be at least several times this minimum perceptible mass thickness difference. Satisfactory replica thickness lies in the range 5–20 μg./cm^2. Since the replica film resolution is about half the *linear* thickness, the resolution is better the greater the density of the film material. Silica has a density of about 2.5, so a film of mass thickness 5 μg/cm^2 has a linear thickness of 200A, and a resolution of about 100A.* To obtain the total resolution, the replica and the instrumental resolution must be added in quadrature. The theoretical resolution of the microscope is about 10A, which is negligibly small in comparison with replica resolution. In practice, due to several factors which cannot be discussed here, electron microscope resolution usually lies between 20A and 50A, and is sometimes no better than 100A. In this last case the total horizontal resolution of a 200A silica film would be about 140A.

3. STUDY OF PARTICLES

In addition to the study of surfaces of various kinds, sizes and shapes of particles form a major field of study. It is perhaps true that most powders of industrial interest either lie completely in, or contain a significant proportion of particles which do lie in, the range of particle size below about 0.5 μ, where ordinary optical methods of examination are unsatisfactory. Among these are the carbon blacks and other fillers used in the rubber and plastics industries, the pigments used in paints, inks and dyestuffs, the clays and other raw materials of the ceramics industry, some of the powders used in powder metallurgical processes, in cosmetics and in pharmaceuticals, to mention a few of the more important. The field of particle study also includes biological particles such as bacteria, viruses, and large protein molecules. A discussion of this last extremely important field cannot be included within the scope of this paper.

A simple way of studying particles consists of dispersing them by mulling in a plastic solution, which is thinly spread on a microscope slide and allowed to dry. The particles are thus cast in a plastic film, which by proper technique can be made amply thin so as to be reasonably transparent to electrons. The particles are more opaque, and so when a micrograph is taken, the particle profiles are seen. Dispersion may also be achieved by other methods such as dusting a dry powder or spraying a liquid suspension onto a thin plastic film, settling from a liquid suspension, use of high frequency (ultrasonic) sound waves, subjecting the powder to action of an intense electric field, and to various combinations of these.†[1,2,3] The supporting membrane is nearly always a thin film

* The reader may have observed that the scale of the micrographs of Figs. 4–10, as given by the micron markers, corresponds to magnification about 6,000. When viewed through a stereoscopic viewer, however, the micrographs are further enlarged about 4×, and total magnification is as large as is useful for micrographs of silica replicas. The half-tone method of reproduction of illustration obviously degrades the pictures greatly, especially when a viewer is used.

In electron microscopy, common practice is to use the Angstrom ($1A = 10^{-8}$ cm.) as a unit of length.

† A method which yields satisfactory dispersion of one powder may not be suitable for another. Detailed descriptions of some of the methods may be found in:

[1] H. C. O'Brien, *J. App. Phys.*, 16, 370, 1945.

of a plastic; and in most of the methods in contrast to the cast-film method the particles are adherent to one side of a film. Figure 13 is a stereoscopic pair showing a carbon black of about 2,500*A* particle size. A microgram and particle size histogram taken from Columbian Carbons 2, p. 37, for a carbon black of particle size about 270*A* is shown in Figure 14. Obviously many of these small particles are partially transparent, and the degree of contrast attainable affects the experimental determination of particle size. Most carbon blacks, at least in the smaller size ranges, tend to have nearly spherical shape, but frequently

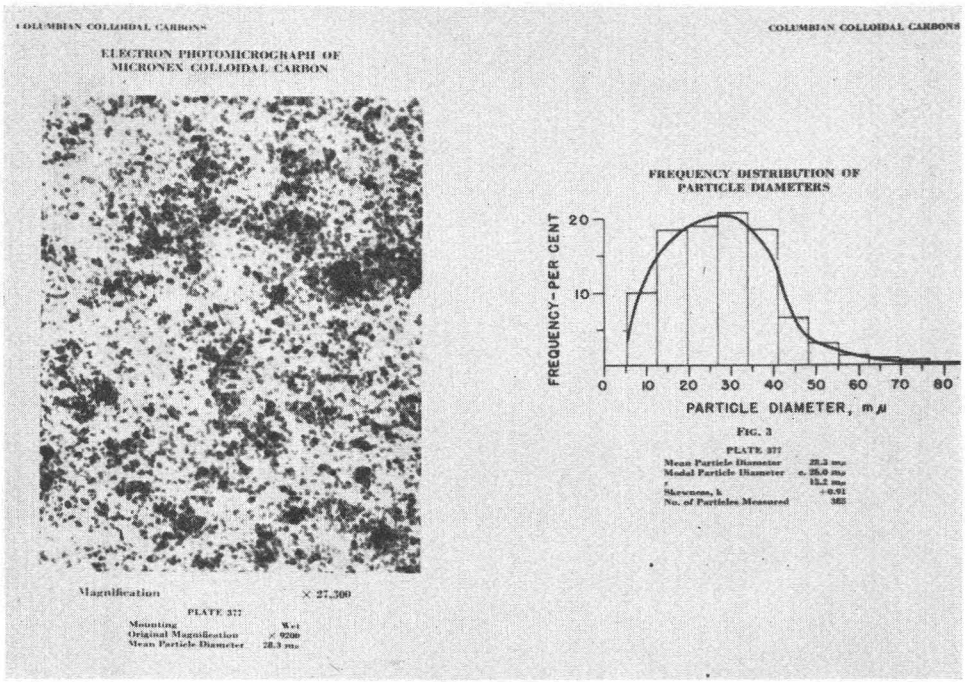

FIG. 14. Micrograph and particle size histogram taken from Colloidal Carbons 2, published by Columbian Colloidal Carbons.

show a reticulate chain structure, in which one particle is joined to another by a cylindrical bar.[2,4]

The need for particle size histograms in the rubber industry is so great that a machine[5] has been devised to plot them semi-automatically. The operator has only to adjust two pairs of mutually perpendicular lines until they frame the particle profile in the micrograph. By means of Selsyn motors the separations of the two pairs of lines are added, to yield a mechanical displacement proportional to the arithmetic mean diameter. This is imparted to a carriage which

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[2] W. B. Ladd and W. B. Wiegand, *Rubber Age*, 57, 299, 1945. Many stereoscopic pairs shown as illustrations.

[3] M. C. Schuster and E. F. Fullam, *Ind. & Engg. Chem.*, An. Ed. 18, 653, 1946.

[4] J. H. L. Watson, *J. App. Phys.*, 20, 747, 1949.

[5] E. E. Hanson and J. H. Daniel, *J. App. Phys.*, 18, 439, 1947.

In the oral paper, slides kindly loaned by Dr. Hanson were used to explain operation of the machine. The original article gives a complete description.

drops a steel ball into one of a number of slots in a lucite plate. Each slot thus corresponds to a small range of values of arithmetic mean diameter, and each ball-in-a-slot represents one particle. After balls representing all the particles measured have been dropped, a photograph of the lucite plate is the particle size histogram.

In the study of particles of various materials, one encounters a great diversity of shapes. Also, it is very difficult to completely disperse the particles, and in some cases there is considerable evidence of fragmentation of the particles during dispersion. These problems are amenable to by the thin-shell silica replica technique, employing stereoscopic pairs for observation. In this method, the particles are dusted onto a microscope slide which has been previously coated with a plastic film. The slide is then mounted in an evacuated bell-jar and silica deposited at an angle by evaporation from a heated tungsten wire conical coil. The evaporating unit with the angle of incidence 60° from the normal to the microscope slide surface is shown in Figure 15. The silica deposits in some such fashion as is shown in Figure 16, about half the incident molecules remaining where they strike the surface, and the other half diffusing over the surface. The bell-jar is then let down to air, the microscope slide turned end for end, and a second similar deposition of silica carried out. The shadow due to the second evaporation is thus on the opposite side of the particle. After removal from the bell-jar, the plastic film is stripped from the microscope slide, placed on a piece of fine mesh screen, and then immersed in a solvent for the plastic. This leaves the silica film with particles embedded in it, supported by the fine screen. It is now immersed in a solvent for the particles, leaving only the silica as a thin-shell replica of the particle surface.

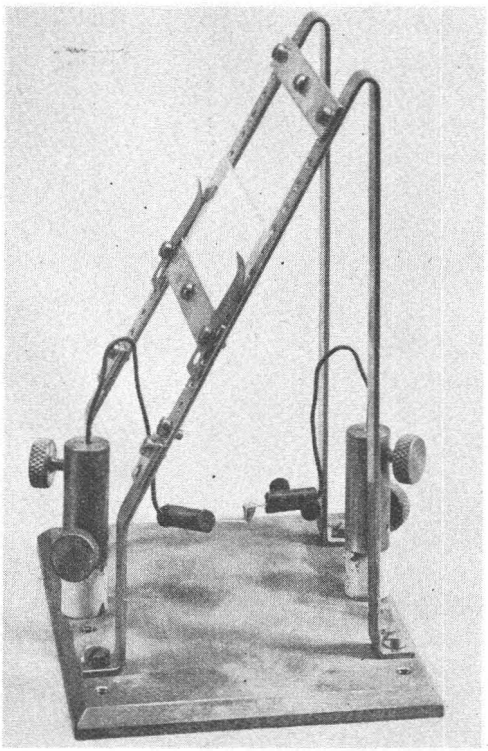

FIG. 15. Experimental arrangement for evaporating silica or silicon monoxide. A few milligrams of the material is placed in the conical toroidal "basket" made of tungsten wire; it evaporates when the basket is heated in vacuum to about 2,000°C. Various supporting frames may be screwed to the base plate; the one shown provides an angle-of-incidence 60° from the normal to the surface of the microscope slide for the silica condensing on the slide.

The next three figures are stereo-micrographs of thin-shell silica replicas of particles. Figure 17 is of a single octahedral crystal of nickel produced by the carbonyl process. One of the shadows is pointed, corresponding to a corner of the triangular top surface; the other is straight, corresponding to an edge. From measurement of the edge length, and the distance between the two shadow edges, the vertical height of the octahedron may be determined. This height can of course also be measured stereoscopically. It is found to agree with its theoretical value of 0.82 times the edge length. The principal uncertainty in the measurements is due to the slightly rounded corners of the crystal.

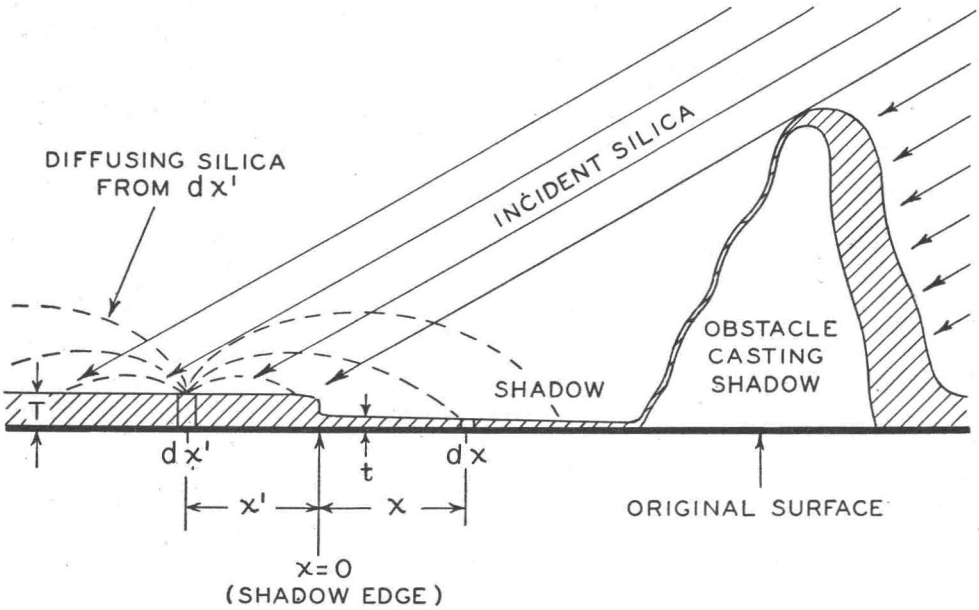

FIG. 16. Diagrammatic representation of the process of condensation of silica. Some atoms stick where they strike and give rise to a shadow edge. Others diffuse over the surface, so that a silica film of lesser thickness is formed in shadowed regions.

As a determinant of vertical heights, the shadowing method is inherently capable of greater accuracy than the stereoscopic, provided that the shadow is cast upon a flat surface, and that the profile which actually casts the shadow can be ascertained, as in Figure 17. This is because horizontal resolution is always much better than vertical, as previously discussed. Furthermore, dependent on the angle of incidence, the horizontal length of the shadow may be

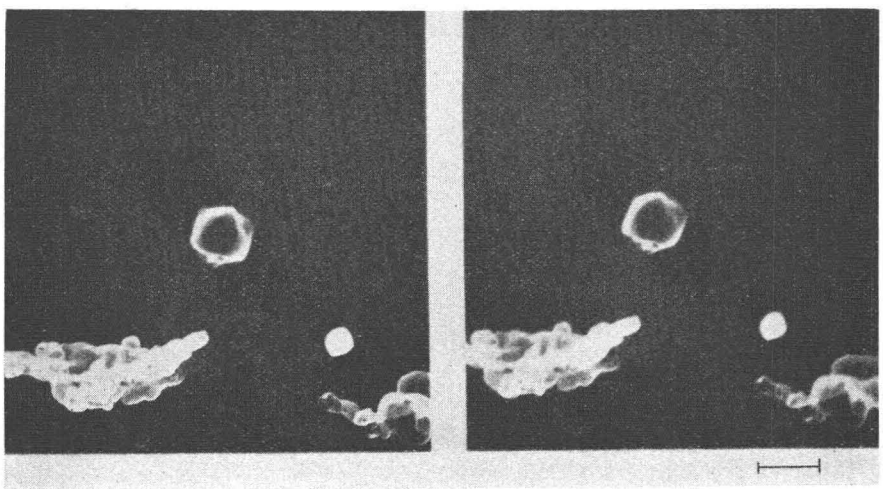

FIG. 17. Thin-shell replica of octahedral crystal of nickel. Double shadows were formed by evaporating silica first from one side, then from the other. Within one of the shadows, silica film has cracked. Cracks like this are due to action of surface tension forces during processing of the replica, and are most likely to be found in the very thin film in the shadows. Angle of incidence of silica was 60° and lengths of shadows determine vertical height of octahedron.

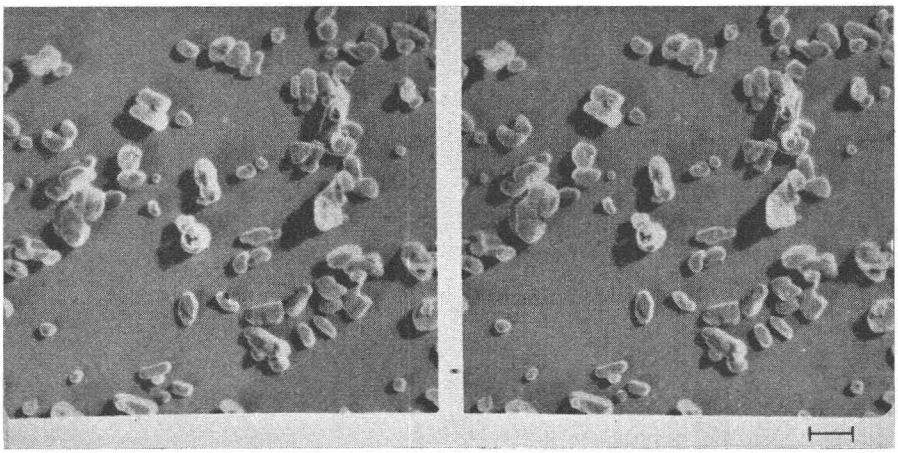

FIG. 18. Thin-shell replica of particles of an alkaline earth carbonate powder. Although double-shadowed, one shadow is very faint due to use of different amounts of SiO_2 in the two evaporations. The observed wrinkling of the supporting base probably occurred during processing and must be taken into account when shadow lengths are used to determine particle heights.

greater than the vertical height of the shadow profile. Magnification factors of this type as great as 5 or even 10 are not uncommon in electron microscopy. However, stereo-micrographs are essential if one wishes to determine whether the two conditions are met. The first condition, for example, may be violated by the surface being rough where the shadow falls, or by its being tilted. Confidence in measurements made on shadow-cast replicas is greatly enhanced by stereoscopic observation, which permits measurement of the three-dimensional topography, sometimes resulting in correction factors to be applied to the observed lengths of shadows.

Particles of an alkaline earth carbonate powder used in making cathodes for vacuum tubes are shown in Figure 18. The shapes of the particles are well delineated, and the shadows provide measures of the vertical dimensions. Further, in many cases of agglomeration, it is possible to distinguish between

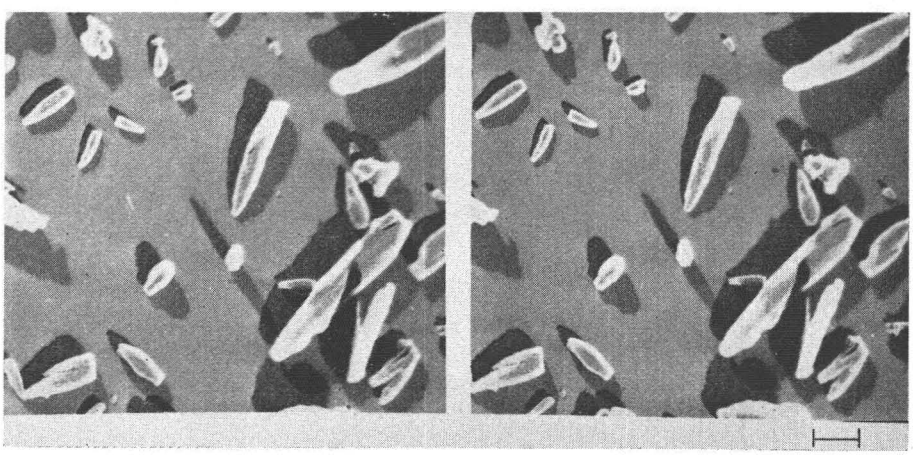

FIG. 19. Thin-shell replica of particles of another alkaline earth carbonate powder. Particle surfaces are wrinkled, and general forms are prolate. Particle near center is long and thin, and resting on a flat end (probably a fragmented section).

individual particles. The last pair, Figure 19, is of particles of a different carbonate. Near the middle of the picture there is a particle nearly standing on end, obviously a long narrow particle. Seen non-stereoscopically it appears roughly egg-shaped. Stereoscopic pictures such as this contain a great deal more

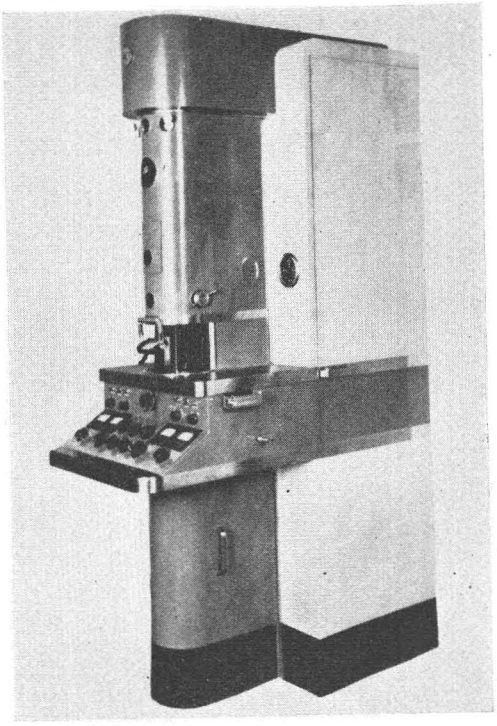

FIG. 20. RCA Electron Microscope, type EMU.

information about each particle than its simple profile. The degree of roughness of the particle surface, its general shape and habit of growth, fragmentation—for example, the central particle has fairly obviously been broken, and is standing on a flat end due to the fracture—the packing of particles in agglomerates, all these factors are susceptible to study and measurement.

It would be possible to go on from here into a discussion of various statistical parameters and their significance in particle study. These include arithmetic and geometric mean diameters, volume- and surface-equivalent diameters, Feret's statistical diameter, and others, which have been developed in attempts to explain the gross properties of powders. But this must await another occasion.

In conclusion, Figure 20 is a picture of the R.C.A. electron microscope at present in use at Bell Laboratories.

I hope I have been successful in presenting to you the electron microscope —and in particular stereoscopic electron micrography—as a new tool of great potentialities in picturing the sizes and shapes of surfaces and particles on a scale comparable with that of the atoms and molecules of which all matter is composed.