

The Thickness of Electron Microscopic Objects*†

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THIS is a preliminary account of experiments made while seeking a way to determine the heights, or thicknesses, of certain objects under the electron microscope. At first, attempts were made to apply the methods of stereoscopic photography to this problem but these were complicated by phenomena presumably due to electron diffraction in the sample. It has been known for some time that effects due to coherently scattered electrons can be recognized under the microscope;

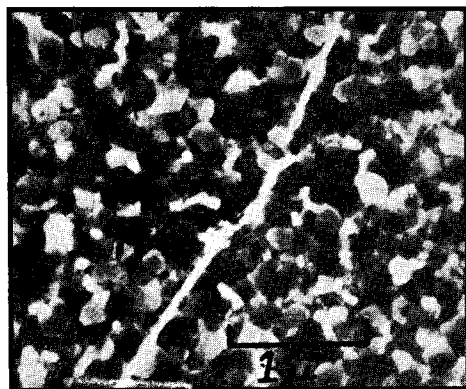


FIG. 1. An electron micrograph made through an evaporated film of metallic bismuth of *ca.* 600A computed thickness. If this film were photographed with the electron beam incident at a different angle (as in a stereoscopic holder) many of the granules which here appear light would undoubtedly be dark, and *vica versa*. Several of the granules in this picture also show the same kind of light and dark lines, or bands, described by von Ardenne, Hillier and Baker, and others.

our results indicate that at times they are of overwhelming importance in determining the appearance of some crystalline objects. Partly because of these diffraction phenomena it soon became evident that information was needed in our work other than that provided by stereoscopic photography. A procedure for measuring

heights based on shadow-casting was accordingly developed to meet this need.

Some months ago Picard and Duffendack¹ published in this journal electron micrographs of several evaporated metallic films. Their photographs of aluminum and copper, for example, consist of an unbroken array of mottled areas, some light and others opaque. Extraordinarily fine pictures of this sort, taken through films of silver and of aluminum by Hass,² have just come to our attention. Bismuth (Fig. 1) in a computed thickness of about 600A also provides a good example of such a micrograph. Such photographs as these have sometimes been thought to indicate that the films themselves consist of granular aggregates of particles of various thicknesses. Early in our work we made stereoscopic photographs of metallic films and were surprised to find that very different detail appeared on the two members of a stereoscopic pair. This is clearly evident in the stereoscopic photographs of an aluminum film reproduced in Fig. 2. If the large irregular object on the left side of the pictures is used as a point of orientation, study shows that very few of the black dots on one stereogram are to be found on its mate. Films of other metals have yielded similar results though failure to match is not always so complete as in this case.

The work that has been done indicates that in general thin metallic films are of three sorts: (1) continuous, with no indication of structure, like chromium, uranium and very thin antimony; (2) continuous, but a mosaic of light and dark patches, like the bismuth and aluminum films of Figs. 1 and 2; (3) a discontinuous assembly of more or less globular-appearing particles, like indium (Fig. 3). Some metals form films of each type depending on thickness and conditions of deposition. As would perhaps be expected, neither member of a stereoscopic pair of photographs of a continuous chromium film shows any

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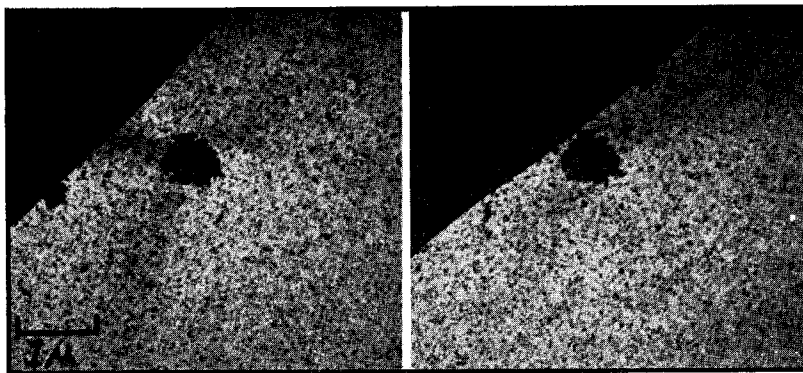


FIG. 2. A stereoscopic pair of electron micrographs of an aluminum film *ca.* 200A thick. The opaque region at the upper left, and the black greatly displaced object on the edge of this opacity, are parts of a wire of the supporting grid (and hence far from the plane of the rest of the preparation). Very few of the dark grains on one photograph appear on the other.

detail whatsoever. Photographs of thin indium (Fig. 3) or of thin bismuth show the same detail on both members of a pair. In such micrographs the individual metallic particles stand out distinctly, but their opacity is not necessarily proportional to their size. The larger particles of thicker films frequently display internal details which are different on the two members of a stereoscopic pair of photographs, and when the film has become so thick as to be substantially continuous it commonly gives a mottled picture like Fig. 1 with features that depend on how the specimen is oriented towards the electron beam.

Much work has been done by electron diffraction on thin metallic films.³ Comparison with these results makes it clear that the films with which we have dealt were crystalline. Without doubt, therefore, the phenomena causing failure to match are due to electron diffraction within the sample. von Ardenne,⁴ Heidenreich,⁵ Hillier and Baker,⁶ Hass,² Kinder,⁷ and others have described effects on electron micrographs due to such diffraction. They are important to all phases of electron microscopy since because of them the apparent opacity of an object under the microscope is not necessarily an index of its thickness. These effects have been of various sorts and have influenced in varying degrees what is seen under the electron microscope. Heidenreich,⁵ for instance, has observed on up-turned crystals of MgO a series of light and dark bands which appear to be due to multiple reflections of the incident electrons, alternately

out of and then back into the path of direct transmission. Kossel⁸ has offered a compatible interpretation of similar photographs by Kinder.⁷ Hillier and Baker's⁶ experiments showed several black and white lines whose sequence and position varied with the orientation of the sample. Similar sets of lines can be seen on other published photographs,^{1,4} notably Hass'² picture of a silver film. His photographs, however, like ours indicate that diffraction effects are not limited to such striations but can cover wide areas. It is evident from our stereoscopic photographs that, especially with some crystalline films, so much radiation may be diffracted that it, rather than the non-coherently scattered electrons, will determine the resulting picture. How completely the appearance of an object under the electron microscope may depend on

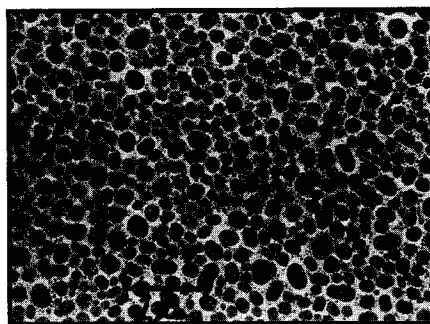


FIG. 3. A photograph through an indium film *ca.* 300A thick. A bismuth film of similar thickness would look almost exactly like this. Here the smallest globules and the big granules appear about equally opaque.

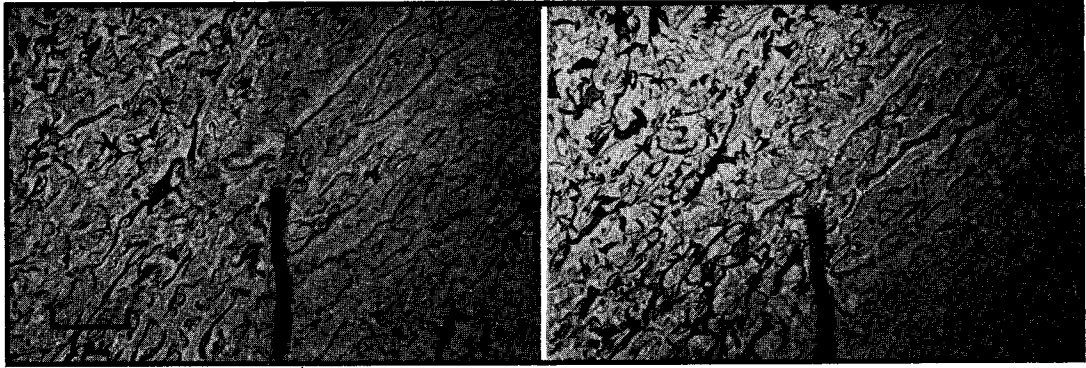


FIG. 4. A stereoscopic pair of electron micrographs made through a film of antimony about 200A thick. Similar regions on the two photographs can be identified by reference to the elongated foreign object in the bottom middle of the field. Successive pictures have been made after an interval of time and after exposure of the film to the air to ensure that the different appearance of these photographs does not arise from changes in the film itself while the experiment was being made.

diffraction is well illustrated by photographs of antimony films. The pair of Fig. 4 was made through a moderately thin antimony film; entirely different regions of darkness, due presumably to the fact that each tilt of the specimen brings different crystalline areas into position for a coherent reflection of electrons away from the transmitted beam, are seen on the two photographs. Even greater difference is shown by the pair of Fig. 5, prepared from a thicker film of antimony; here one knows he is looking at the same field only because identifying holes can be found in the two horizontal cracks across the lower part of the pictures.

It is evident that this dependence of electron microscopic appearance on specimen orientation cannot always be recognized or fully evaluated by studying a single photograph; it is equally clear that other sorts of crystalline materials than metallic films will show these effects. As a result of our experience we would hesitate to interpret

in terms of thickness or density any detail on an electron micrograph without studying photographs made at more than one angle of tilt.

The unsatisfactory nature of stereoscopic observation as a way of measuring the heights of certain electron microscopic objects leads us to propose another method based on shadow-casting. It consists essentially of depositing a thin metallic film obliquely onto the preparation in question and then photographing and measuring the lengths of the shadowed areas thus formed on the preparation. There has just come to our attention a paper by Müller⁹ who describes a similar shadow technique. Our procedure differs from his in two respects: (1) in the choice of a metal for evaporation that will yield a very homogeneous film, will show no tendency to migrate over the substrate, and thus will yield sharp, clearly-defined shadows; and (2) in casting two shadows simultaneously from directions about 180° apart (Fig. 6). The experi-

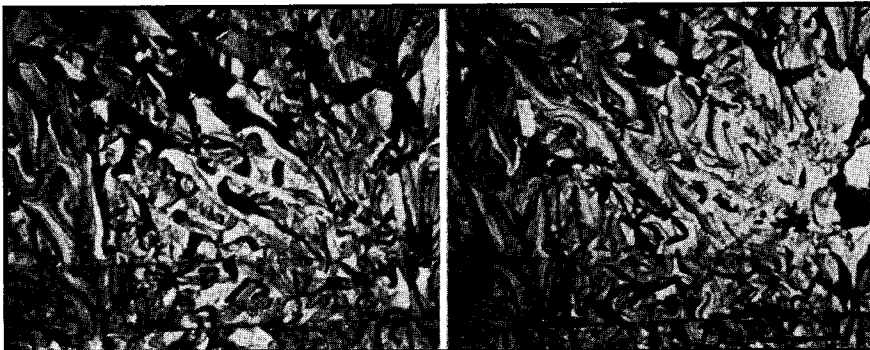


FIG. 5. A stereoscopic pair of micrographs through a thicker antimony film (computed thickness *ca.* 400A).

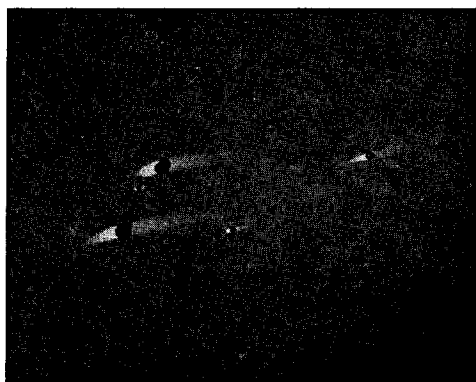


FIG. 6. Photographs of the shadows obtained when thin films of chromium are evaporated from opposite directions onto a collodion film supporting certain biological objects. The two filaments from which evaporation took place were at different heights above the preparation so that one set of shadows is always longer than the other.

mental arrangement needed for shadow-casting is the simple one, familiar to all who have worked with evaporated films, shown diagrammatically in Fig. 7. Metal from two hot filaments is evaporated simultaneously, or successively, at known angles onto the specimen suitably mounted in the middle of an evacuated bell jar. Two opposite shadows are required since the specimen on its screen is often not horizontal and this will of course greatly alter the lengths of the individual shadows. A sufficient accuracy will, how-

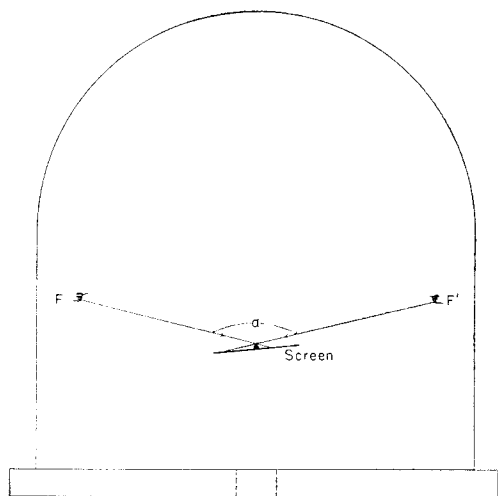


FIG. 7. A schematic drawing of apparatus for shadow-casting onto electron microscopic preparations. The dark object shown in the center of the screen protects portions of the screen from atoms evaporating from filaments F and F' , and thus creates regions that appear like shadows.

ever, result by using the average length of opposed shadows. It is obvious that this method cannot be applied to objects so shaped or situated that they do not cast shadows and that it has only a limited accuracy for many irregularly shaped objects—but for all recognizable objects it can set an upper limit to the height and for many that do cast shadows it sets both an upper and a lower limit. It is practical to work with shadows cast at angles such that the length of shadow is five to ten times the height of the object. Chromium and uranium are useful as shadow-casting metals since both deposit in uniform, structureless films. Uranium will cast perceptible shadows in computed thicknesses of only 5–10Å; with chromium about 50Å films are needed. As yet we do not know the minimum height that can be measured in this way but we have seen sharp little shadows (Fig. 8) whose lengths indicate that the objects responsible for them were not more than *ca.* 30Å high. This is definitely less than the resolving power of the microscope under the conditions prevailing when this picture was made. Evidently shadow-casting can be used for two purposes: (a) to estimate the heights of objects seen under the microscope and (b) to extend still further the limits of our observation giving some idea of the existence and conformation of things below the resolving power of the microscope. Since shadow-casting makes it possible to estimate thicknesses in the

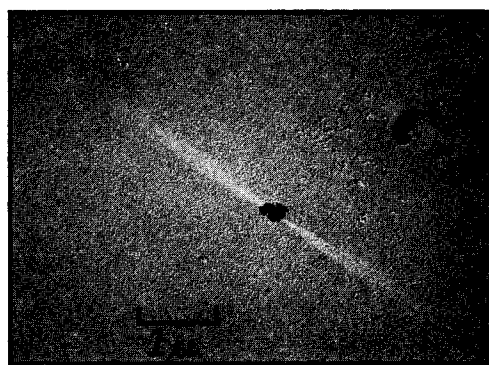


FIG. 8. Another example of a shadow-cast electron micrograph for the determination of the heights of objects. Close inspection of the picture shows minute, sharp shadows down to the limit of visibility to the eye. Shadows cast here are seven times as long as the objects are high. It is to be noticed that in this, and in Fig. 6, the shadow-casting technique brings out much detail of the structure of the supporting collodion films.

range of molecular dimensions, it opens up an interesting field of experiment.

BIBLIOGRAPHY

- (1) R. G. Picard and O. S. Duffendack, *J. App. Phys.* **14**, 291 (1943).
- (2) G. Hass, *Kolloid Zeits.* **100**, 230 (1942).

- (3) G. P. Thomson and W. Cochran, *Theory and Practice of Electron Diffraction* (The Macmillan Company, London, 1939).
- (4) M. von Ardenne, *Zeits. f. Physik* **116**, 736 (1940).
- (5) R. D. Heidenreich, *Phys. Rev.* **62**, 291 (1942).
- (6) J. Hillier and R. F. Baker, *Phys. Rev.* **61**, 722 (1942).
- (7) E. Kinder, *Naturwiss.* **31**, 149 (1943).
- (8) W. Kossel, *Naturwiss.* **31**, 323 (1943).

Potential Nuclear Monokinetic Electron Sources*

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In this paper sixty-eight nuclear monokinetic electron sources are tabulated. These sources are obtained by producing designated artificial radioactive elements. The voltage of the electrons emitted in the lowest energy group is 17 kev and that in the highest is 578 kev. Applications seem possible in special radio tubes and for certain experiments with electron microscopes and electron diffraction.

AMONG the some 300 synthesized or artificial radioactive nuclei discovered to date, a few have been found which emit electrons in a narrow energy range. Most nuclei, however, emit the well-known and characteristic continuous beta-ray spectrum of electrons or positrons or both.

A summary of the available nuclei capable of emitting monokinetic electrons is given in Table I. The next-to-the-last column lists the energy of the emitted electrons in kilovolts in the order of increasing energy. As may be seen the lowest energy group of electrons, No. 1 of the first column, comes from tungsten of atomic mass number 187. The highest energy group, No. 68, comes from tellurium of atomic mass number 122 or 124. The half-life of this emitting nucleus is 125 days. The minimum and maximum energies are, respectively, 17 and 578 kilovolts.

In most cases only a rudimentary knowledge is available about the monokinetic sources. In a few cases, however, a reasonable amount of detailed work has been reported by the use of a beta-ray spectrometer¹⁵ or spectrograph.² In these instances two monokinetic groups are observed, one which is associated with the *K*-electron shell of the atom and another which is associated with the *L*-electron shell. The group

associated with the *K*-electron shell is in general the stronger by a factor of eight times. The factor, nevertheless, varies considerably from element to element; for No. 20 the two groups seem to be of equal intensity. In Table I only the monokinetic groups associated with the *K*-electron shell are listed in the next to the last column.

For a given element the energy difference between the two emitting groups is equal to the energy of the $K\alpha$ x-ray line of the element. This value varies from 3.7 kilovolts for No. 55 to 91 kilovolts for No. 19. Sometimes an electron group has been found which can be identified with the *L* series, but in every case the intensity is very weak.

Contributions to the theoretical significance and interpretation of the origin of monokinetic electron groups have been made by a number of authors.⁴⁵ The probability of a gamma-ray being internally converted, the magnitude of the *K* to *L* conversion ratio, and the function relating the conversion ratio with the half-life periods and the energy states of nuclei are still in the formative stages.

To predict that nuclear monokinetic sources will be of practical importance in the near future is, at present, without adequate foundation. The state of development is now remindful of that of the Edison effect in 1883. There are a few nuclear sources which even now can be made to

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