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THE ELECTRON MICROSCOPE AS AN AID IN COLLOID RESEARCH (DAS ELEK--ETC(U)
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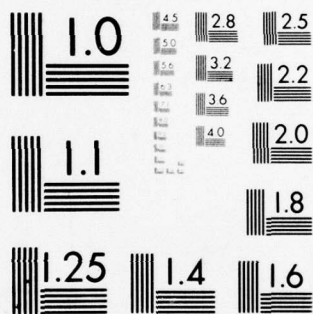
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The results achieved previously with the magnetic electron microscope permits it to be surmised that this instrument will also be a useful instrument for colloid research. According to definition this branch of natural science includes our knowledge of the physical and chemical properties of material aggregates which have measurements at least in one direction between about 1 and 500 μ .

Because most of the properties of colloidal systems depend closely upon the size and shape of the aggregates, their evaluation is almost always a prerequisite to any work with these systems. The light microscope, whose resolving power in oblique illumination with visible light is about 160 μ , can be used with ultraviolet light (resolving power about 80 μ) in the study of coarse colloidal systems at the expense of much care and equipment. However, special procedures must be developed for evaluating sizes in the range of the most important colloidal systems. It is impossible here to evaluate the advantages and disadvantages of most of these systems (2,3). Altogether, it can be said that the size of colloidal particles can be determined in most cases satisfactorily from properties toward light (ultramicroscopy, Tyndallometry, turbidity measurements) or by movement phenomena (diffusion and sedimentation measurements, viscometry), or by ultrafiltration.

On the other hand, for the evaluation of shape, or geometric form, only a few methods are available. Thus we are able to make justified statements about the form of colloidal aggregates only in particular cases. Mostly, it

can be determined only whether particles are spherical or anisodimensional, which is to say, whether in a given instance one or two dimensions are greater than the others, without being able also to draw conclusions as to the size of the single dimensions. Thus, from the "flickers" of the particles in the ultramicroscope, or beyond this, from the state of polarization of the Tyndall light and the streaming birefringence the presence of anisodimensional particles can be ascertained. Furthermore, in certain instances quantitatively permissible statements can be made about the dimensions of colloidal particles from the scattering of the DEBYE-SCHERRER lines.

Because the use of the procedures given is only limited, and because the significance of the experimental results is frequently burdened with a series of preconditions that are difficult to control, a direct visualization procedure, like the microscope is for larger particles, would be very desirable in colloid research. In what follows it will be shown that in some systems the electron microscope, although it is still in the beginning of its development, already can yield really rather informative images of colloidal aggregates.

1. Investigation of a system with nearly cubical particles

Initially, the pyrosol* ruby glass, which was one of the first objects

* Pyrosols are colloidal dispersions in a solid dispersion medium (2 and 5).

of study with the ultramicroscope by ZSIGMONDY and SIEDENTOPF (6), was selected as an example. Because gold dispersions in borax glass are easier to prepare than those in silicate glass, we studied a borax pyrosol. The mean gold particle size in borax melts prepared in various ways have been

calculated by EHRLINGHAUS and WINTGEN (7) from the gold concentration and the number of particles estimated with the ultramicroscope. For example, in a light blue gold borax glass with 0.012% gold content they find a mean particle size of 120 mp. By blowing a similar melt with compressed air, we obtained very thin flakes, of which Fig. 1 shows a segment in the electron image. Because with the electron microscope using a sufficiently great electron velocity the contours in the image of the irradiated objective are caused principally by differences in the irradiated mass density, or that is, by the product resulting from the irradiated path and relative density, the flakes must be made as thin as possible.

The very thin walls of the bubbles, which we obtained according to the method of producing soap bubbles, fulfilled this requirement. The gauging of the gold particles in the electron image, which here as in all of the subsequent cases were each put on the original plate shows particle sizes that range between 100 and 150 mp. In addition to the particle size the degree of dispersivity can be estimated from the electron images just as well. Because the particle images are more or less round in shape, it can be concluded that no one dimension in the form of the gold particles of the pyrosol is essentially greater than the others.

Gold pyrosols in lithium borate were also made. Because lithium borate is even better irradiated by electrons than sodium borate (borax), the gold particles in these images can be seen very readily. If lithium borate, prepared from one part lithium carbonate and two parts boric acid, is blown quickly at the correct instant during cooling, very thin films can be obtained that can be used as a "microscope slide" in electron microscopy.

However, microscope slides from organic material can also be used.

Fig. 2 shows gold particles on a thin gelatin film. During the manufacturing of this preparation an alcoholic gold solution, which had been obtained by burning an electric arc under alcohol for 10 days after the preparation, was dropped onto the slide. The sol was a blue violet color. By means of the ultramicroscope a particle number of 9×10^7 particles/ml was measured. The gold concentration of the sol was 2.2×10^{-6} g/ml. From this it was possible to estimate the mean particle size as 65 mp. To judge by the varying luminosity of the diffraction images in an ultramicroscope, particles of various sizes were present in the sol. When the alcohol was evaporated the drops clumped together on the gelatin film, leaving behind mainly coarse gold particles (in the lower part of the image). On the average, these particles have a size of 100 mp. The finer particles in the upper part of the image which remained behind after the drops were completely evaporated, have an average size of 50 mp. For this example the preparation of the sol according to the procedures of the unshielded direct current arc discovered by BREDIG in 1898 was deliberately selected in order to show how easily the degree of dispersivity can be determined from the electron images. Additional statements regarding the form of the particles can be made from the image. Most of the particle images measure almost twice as much in one dimension as in the others. A definitely uniform orientation of the long axes of the particles (from top to bottom in Fig. 2) thus is probably due to the forces acting on the rim of the evaporated drops. The fact that colloidal gold particles are not only in modular form, but also flakes or rod form is well known, and already has been the object of many studies. Details of this can be obtained from the monograph of ZSIGMONDY and THIESSEN (8) on colloidal gold. It has given

especially satisfactory information about anisodimensional particles in the study of streaming birefringence and birefringence in an electric and magnetic field. With the help of the electron microscope it could be easily possible to learn which assymetry in shape is sufficient to induce streaming birefringence.

2. Investigation of a system with filamentous particles

During the thermal decomposition of iron pentacarbonyl and nickel tetracarbonyl (9) long, very thin filaments of iron and nickel arise (tertiary structures), which are composed of many single sections (secondary particles). Fig. 3 shows in a microphotograph a bundle of these filaments. The single filament sections consist of even smaller, longish crystallites (primary particles), whose existence and size was determined from the scattering of the interferences in the DEBYE-SCHERRER diagram. In nickel the primary crystals are $20 \times 6 \times 4$ μ p size. The iron crystallites have about the same sizes, but here it is not the lll axis, as with nickel, but the $0ll$ axis that exhibits the largest compass. We were able to explain the formation of the filaments in a way where the primary crystallites, which build up during the condensation of the metal fog, are small elemental magnets that mutually attract each other during aggregation and are bonded together directionally into particles of about 100 μ p size (secondary particles). These still carry free magnetic moments and orient themselves together directionally into the filaments observed in the sediments (9).

Because even at higher magnification the photographs of the metal filaments were not able to provide any additional information about the type of bonding together, we have studied whether the position of the primary and

secondary particles can be identified under the electron microscope. As preparations we used sediments which had been deposited on a fine mesh molybdenum grating from the pertinent aerosols. The filaments then lay without support between the meshes of the grating. Fig. 4 shows an electron image of this kind. This picture of iron filaments is one of a series obtained of electron images of iron and nickel filaments which all show that the filaments are not round, but have a ribbonlike structure. Because the iron filaments are still within the field of the objective coil, the bands have a definite orientation. Parallel stria can be made out across the longitudinal axis at relatively regular distances. There are alternating positions of greater or lesser thickness in the structure of the band. The thicker areas, which we regard as secondary particles have a linear form. Their long axis is 100-120 mp, and their two other axes are about 50 mp wide.

It is rather difficult to draw conclusions from the electron image about the position of the roentgenographically measured primary crystallite in the secondary particle, because this crystallite has a size of about $20 \times 5 \times 5$ mp. Fig. 5 shows a light enlargement of one point of the electron image of Fig. 4. A part of a strip that has been well irradiated by electrons is visible, with a narrow transverse stripe and on the edge a serrated formation. These figures are in the size range of the dimensions of the primary crystallite measured in the roentgenographic path. On the upper edge of the strip several parallel streaks are seen to be situated, whose width is between 5 and 10 mp, and whose length is about 20 mp. Long, thin sections of strips like this probably arise when the primary particles immediately form into filaments, without secondary particles first arising. In substance, though, the picture is to primarily indicate only the possibility that perhaps in the future the size calculated from the scattering of the x ray interferences

and the position of the crystallite estimated from the filament diagram can be made directly visible with the electron microscope.

Principally, this example of a filamentous system is to show how well the main characteristics of colloidal aggregates, namely, the size and the geometric form of the smaller parts, as well as their type of overall structure, can be observed with the electron microscope.

3. Studies of systems with parts in the shape of foils

a) Electron image of a metal foil. Fig. 6 reproduces the electron image of a silver foil, which was obtained from an electrolytic precipitate by etching to about 100 mp thickness. The broad, black band going across the picture is from a 33 mp thick wire of the molybdenum wire netting on which the foil was laid. With the aid of this known measure the magnification of the image can be ascertained. At the white spots the foil is completely eaten through by the etching agent.

As is known for precipitates prepared electrolytically, a determinate direction of the crystallite predominates. However, it is not possible to estimate the size of the original electrolytically precipitated crystallite, because it is not known how intensely the single crystallites have been etched away. A picture (Fig. 7) is magnified to show how well the electron microscope is able to resolve fine structures. The distance between centers in the middle of the picture is 45 mp. The other dark areas at the edges are semiparallel lines at 15 mp distance that are still well resolved. In this picture the black spots are the holes in the foil.

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LEGENDS FOR FIGURES

- Fig. 1 Electron image of colloidal gold in borax glass. Particle size 100-150 mp. $U = 79 \text{ KV}$; $V = 2,200:1$
- Fig. 2 Electron image of colloidal gold on gelatin film. Particle size 50-100 mp. $U = 76 \text{ KV}$; $V = 2,900:1$
- Fig. 3 Microphotograph (direct illumination) of a sediment from an iron aerosol. $V = 300:1$
- Fig. 4 Electron image of colloidal iron filaments. Filament width 100-150 mp. $U = 79 \text{ KV}$; $V = 3,100:1$
- Fig. 5 Electron image of a portion of a colloidal iron filament. In the structure of the filament the points seen are separated by a 5-10 mp distance. $U = 79 \text{ KV}$; $V = 3,100:1$; light $V = 9.5:1$, total $V = 30,000:1$
- Fig. 6 Electron image of an electrolytically prepared and then etched silver foil. $U = 77.5 \text{ KV}$; $V = 980:1$
- Fig. 7 Electron image of an etched silver foil. Distance between centers in the middle of the picture: 45 mp. $U = 76 \text{ KV}$; electron $V = 2,500:1$; light $V = 7.5:1$, total $V = 19,000:1$