A METHOD OF EXAMINATION OF SECTIONS OF FINE METAL POWDER PARTICLES WITH THE ELECTRON MICROSCOPE

With 17 figures

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Introduction

The aim of this note is the description of a technique to be applied to the study of sections of metal powder particles, less than 20 microns in diameter, with the electron microscope, by the replica method, using for replica a material such as formvar or parlodion.

This work originated with the study of particles of tungsten powders. Such particles were too small to be examined in sufficient detail with the light microscope. On the other hand, examination with the electron microscope of the powders dispersed in the supporting film was not found satisfactory because—

(a) it did not permit differentiating between single particles and aggregates,

(b) it did not show the true outlines of the particles but only the projection of a whole surface,

(c) no details appeared.

For that reason it was attempted to make replicas of sections of the particles imbedded in a mounting material suitable for polishing and etching. The method proved satisfactory for the examination of tungsten powder and was then extended to carbonyl nickel and carbonyl iron powders.

Procedure

Several mounting materials commonly used in ordinary metallography were investigated. The problem consisted in finding a mounting material that would—

(1) wet the metal particles,

(2) be of proper hardness to give, after polishing, a fairly smooth surface from which a replica could be stripped,

(3) resist the attack of etchants,

(4) be incompatible with the replica material to permit stripping.

Of the mounting materials investigated, aerotex[1]) was found to be the easiest to use. A thick paste was made by mixing the powder in a little aerotex. A thin layer of this mixture was spread on a square of glass, about $^1/_2''$ on the side, thick enough to permit easy holding during polishing. The best final surface was obtained by spreading the mixture of powder in aerotex into a film as thin as possible. Under such a condition, the aerotex polymerizes into a continuous film that seems to gain strength from the glass backing. A thick layer of aerotex gives a porous rough surface after polymerization. Polymerization was carried out in a drying oven at 140° C. for two hours. The specimens were then carefully polished in the ordinary manner through emery papers, silk cloth and velvet, and they were etched. The usual precautions to prevent removal of inclusions from metallographic specimens had to be observed. Polishing required a little care, but it was not difficult. Either formvar or parlodion replicas could be stripped from the surface and shadow-cast as is customary.

Results

Fig. 1—4 show sections through particles of a sample of tungsten powder. The shape and microstructure of the particles are clearly visible. Evidently many aggregates actually consist of small particles sintered together; the sintering probably took place during reduction of the tungsten oxide in the preparation of the metal powder. The resolution power of the light microscope is not sufficient to permit such an observation. Examination with the electron microscope of the powder dispersed in the supporting film also fails to indicate whether the aggregates consist of separate particles just touching one another or of particles bonded together into actually much larger particles.

Fig. 5—8 show sections through particles of a sample of carbonyl nickel powder. In this case, the large specific area of the powder is evidenced by the jagged outline of the particles. Again the resolution power of the light microscope is too low to reveal the surface condition of the particles of such a powder. Examination with the electron microscope of the powder dispersed in the supporting film would also fail to bring out the surface details of the powder or would, at least, show them less clearly than replicas of the sections do.

Fig. 9—17 show sections through particles of a sample of carbonyl iron powder. The shape and microstructure of the particles are again clearly visible. The diversity in grain size and structure of the particles of a small sample of a powder is striking. Fig. 9—12 illustrate differences in grain size; Fig. 13—16 bring out differences in the structure of particles with the characteristic "onion skin" appearance; Fig. 17 is a photomicro-

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[1]) Aerotex — M-3, water soluble, melamine formaldehyde plastic.

graph of the same particle as Fig. 13 at a higher magnification; it emphasizes the presence of two finely dispersed constituents which are alternately in larger proportions forming concentric darker and lighter rings. The number and sharpness of the structural details visible with the electron microscope is greatly increased over those brought out with the ordinary microscope. From observations with the ordinary microscope, for instance, one might be tempted to exaggerate the occurrence of particles with an "onion skin" structure and to attribute the absence of such a structure in a large number of particles to improper etching. Really many particles consist of polyhedral grains of iron too small to be resolved with the light microscope.

Conclusions

The photomicrographs in this paper prove that the application of the electron microscope to the study of fine metal powders can be extended to the examination of sections of the particles. The microstructure of such particles can thus be revealed beyond the range of resolution of the light microscope. Added information can also be obtained on the size, shape and surface condition of metal particles by a study of their cross sections with the electron microscope over the information obtainable with the same instrument from a study of the powders dispersed in supporting films.

Acknowledgement

The author is grateful to Sylvania Electric Products Inc. for its generous support of this work. The author also wishes to extend her thanks to Mr. W. E. KINGSTON, Manager of Metallurgical Research, for his patient guidance, to Mr. G. A. DAVIS, Section Head of Physical Metallurgy, for his assistance and constructive criticism, and to Dr. B. KOPELMAN, Section Head of Basic Research, for his advice on plastics.

Methode zur elektromikroskopischen Untersuchung von Schliffen von Metallpulvern

In dieser Mitteilung wird eine Methode zur elektronenmikroskopischen Untersuchung von Schliffen von Metallteilchen, kleiner als 20 Mikron im Durchmesser, beschrieben.

Die Metallpulverteilchen werden mit Aerotex, einem plastischen Material, gemischt und die Mischung in Form eines dünnen Films auf eine Glasplatte aufgetragen und polymerisiert. Schleifen, Polieren und Ätzen wird in der metallographisch üblichen Weise vorgenommen. Von der Schliffläche werden Abdrucke genommen und im Elektronenmikroskop untersucht.

Die Resultate dieser Untersuchung werden an Hand von Beispielen (Wolfram, Karbonyl-Nickel und Karbonyl-Eisen) gezeigt.

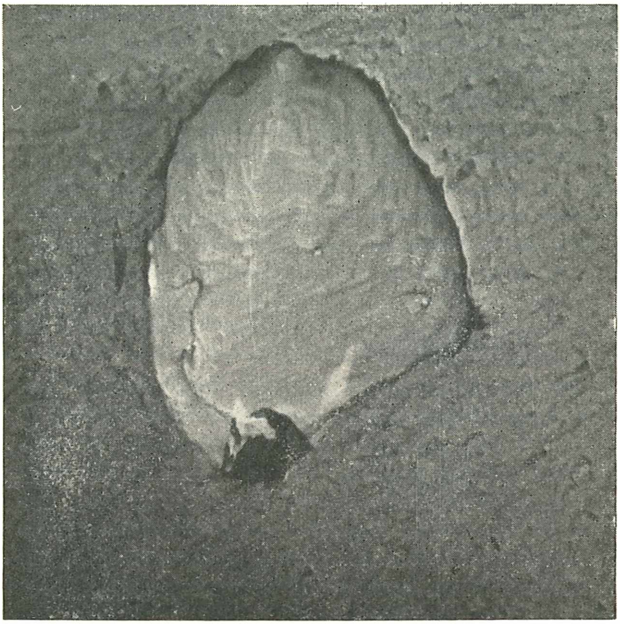


Fig. 1.

Fig. 1 and 2. Tungsten powder particles. 10.000 : 1.

Fig. 2.

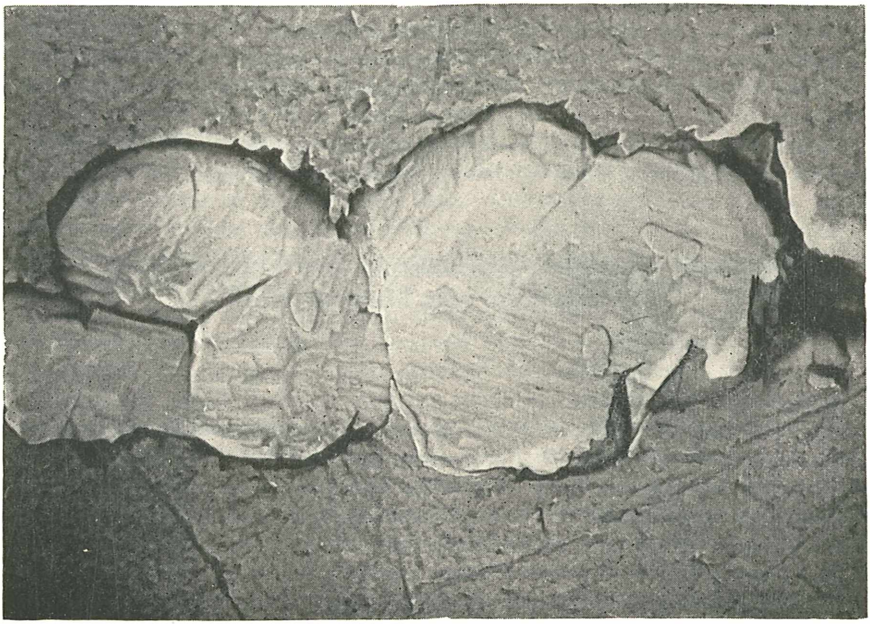


Fig. 3.

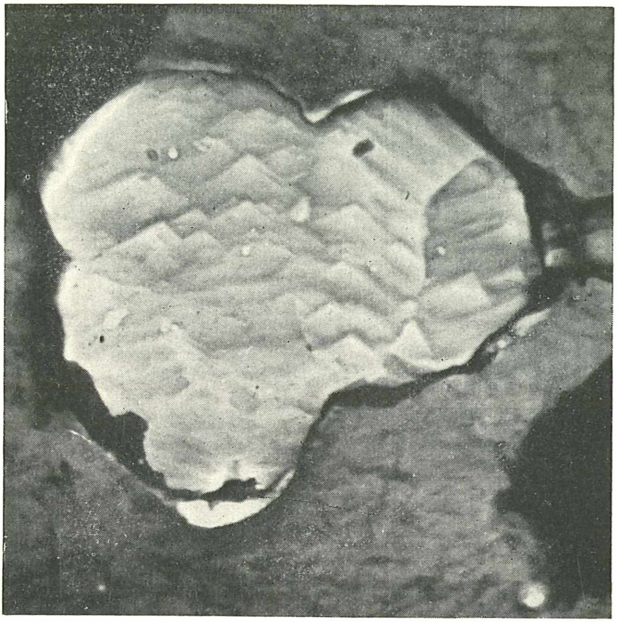
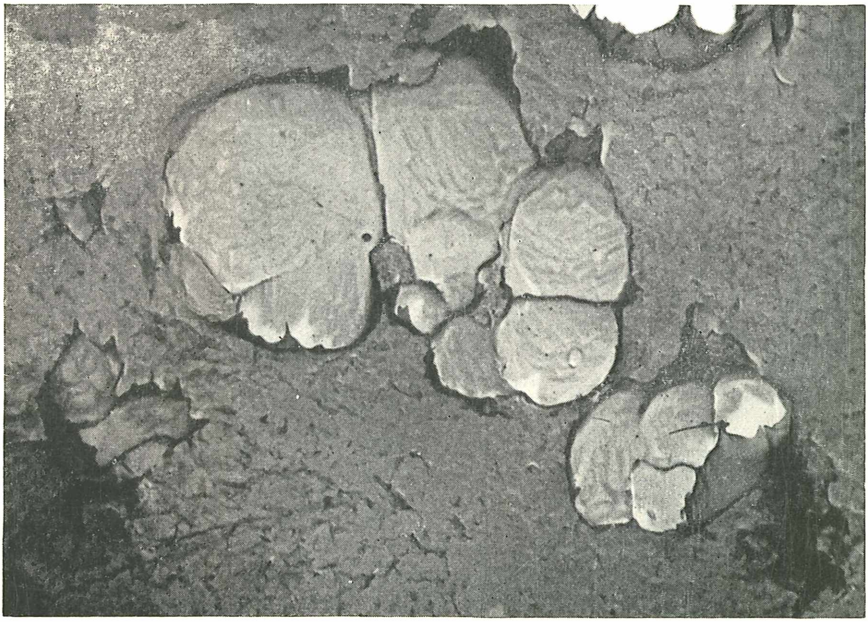


Fig. 3 and 4. Tungsten powder particles. 10.000 : 1.

Fig. 4.



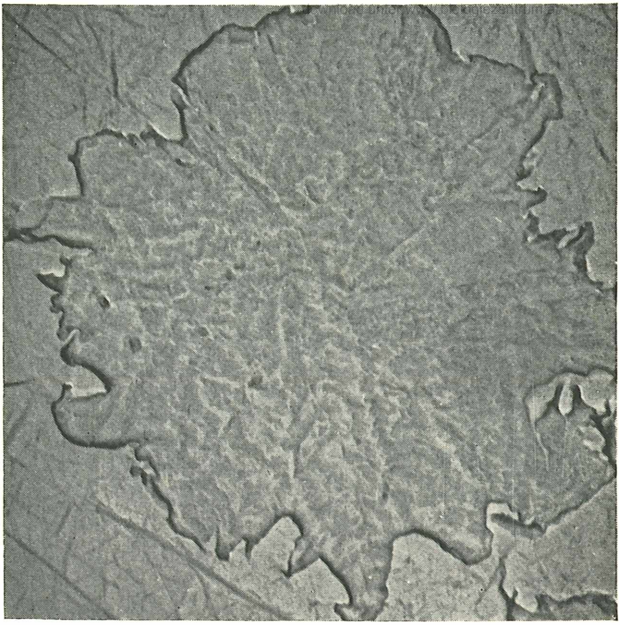


Fig. 5.

Fig. 5 and 6. Particles of carbonyl nickel powder. 10.000 : 1.

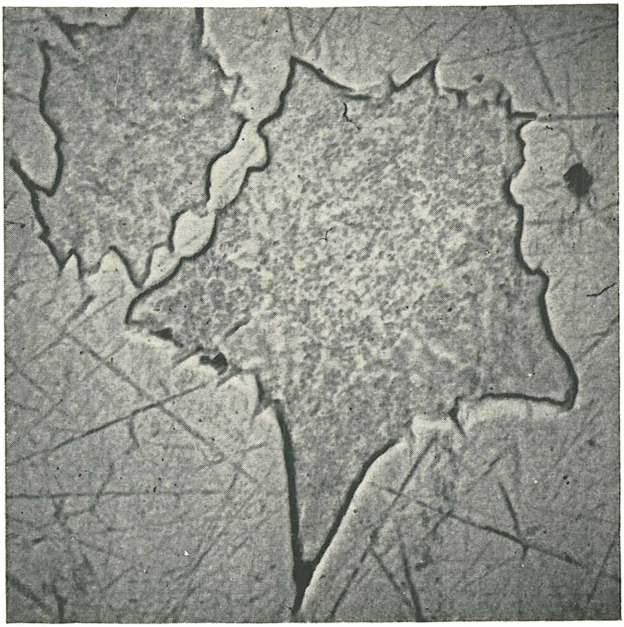


Fig. 6.

Fig. 7.

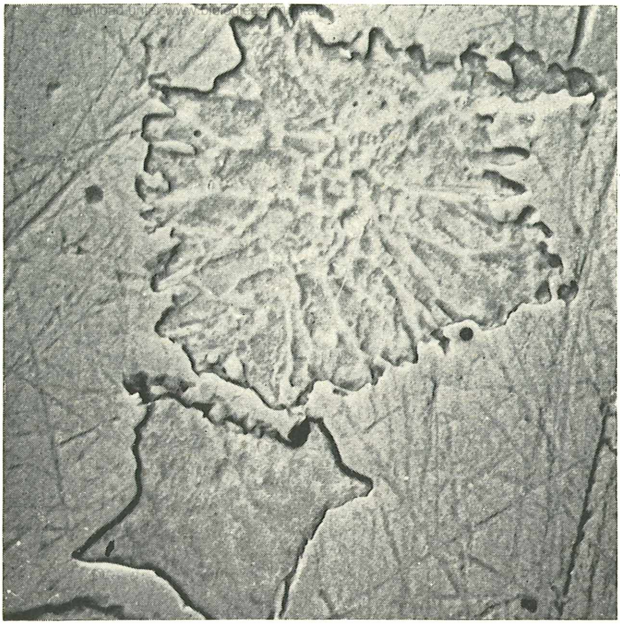


Fig. 7 and 8. Particles of carbonyl nickel powder. Fig. 7, 10.000 : 1. Fig. 8, 5000 : 1.

Fig. 8.

Fig. 9.

Fig. 9 and 10. Particles of carbonyl iron powder. 10.000:1.

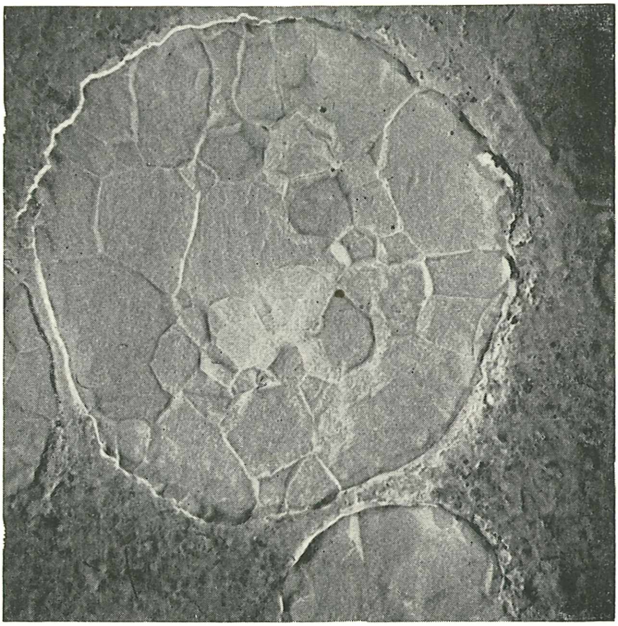


Fig. 10.

Fig. 11.

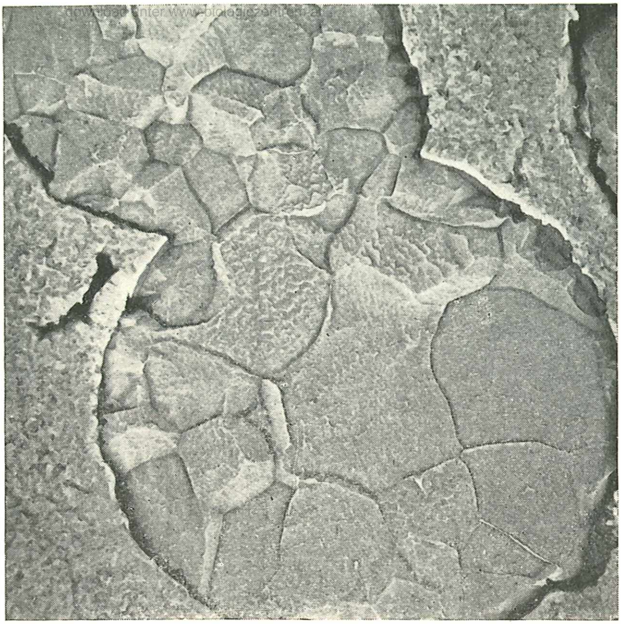


Fig. 11 and 12. Particles of carbonyl iron powder. 10.000 : 1.

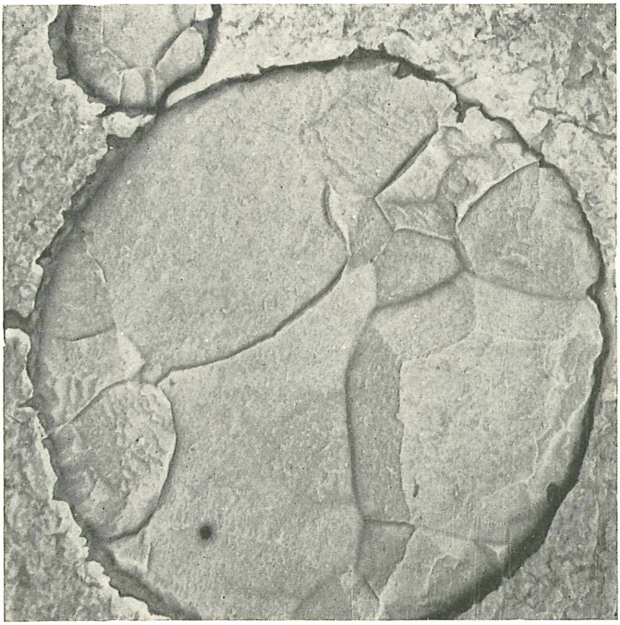


Fig. 12.

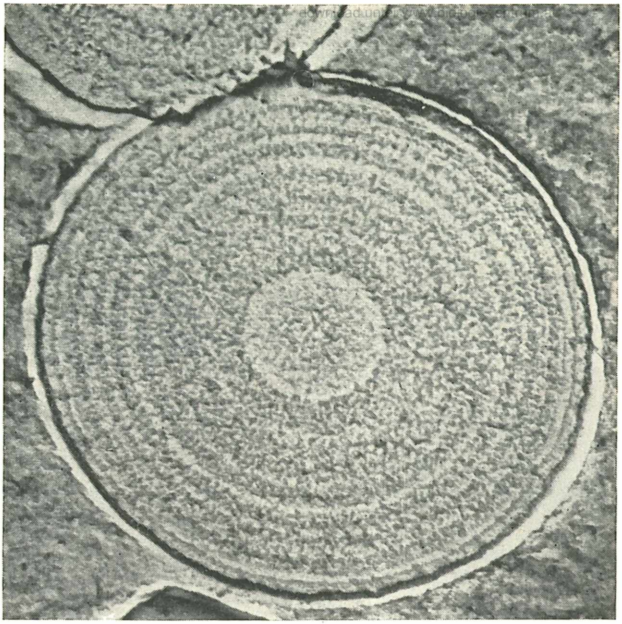


Fig. 13.

Fig. 13 and 14. Particles of carbonyl iron powder. 10.000 : 1.

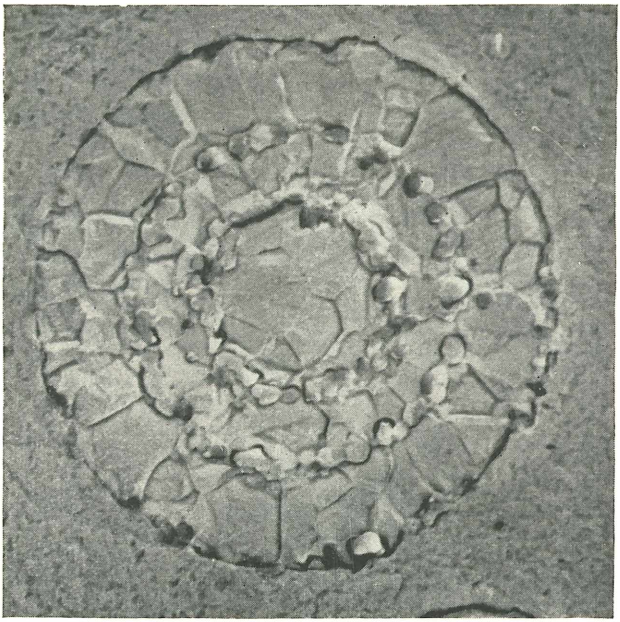
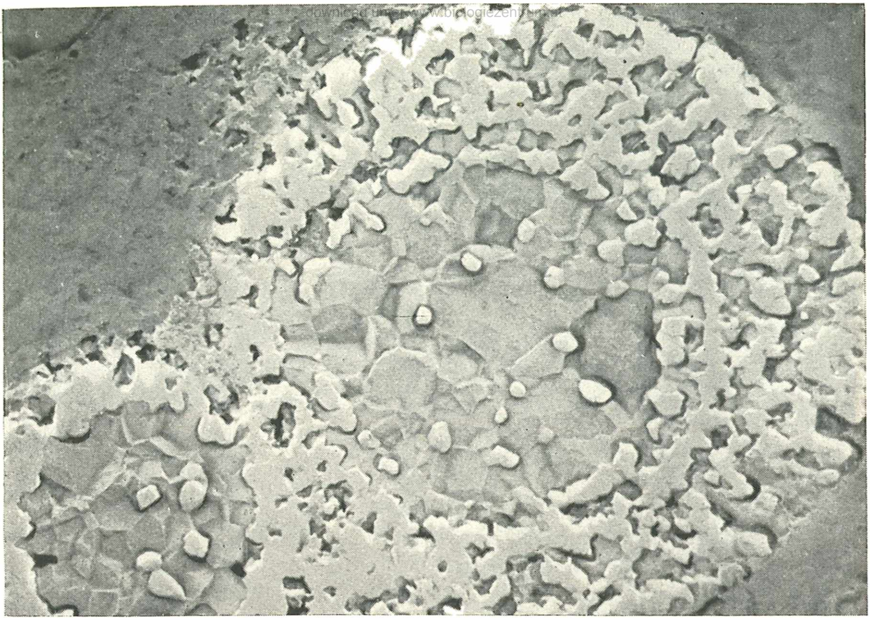


Fig. 14.



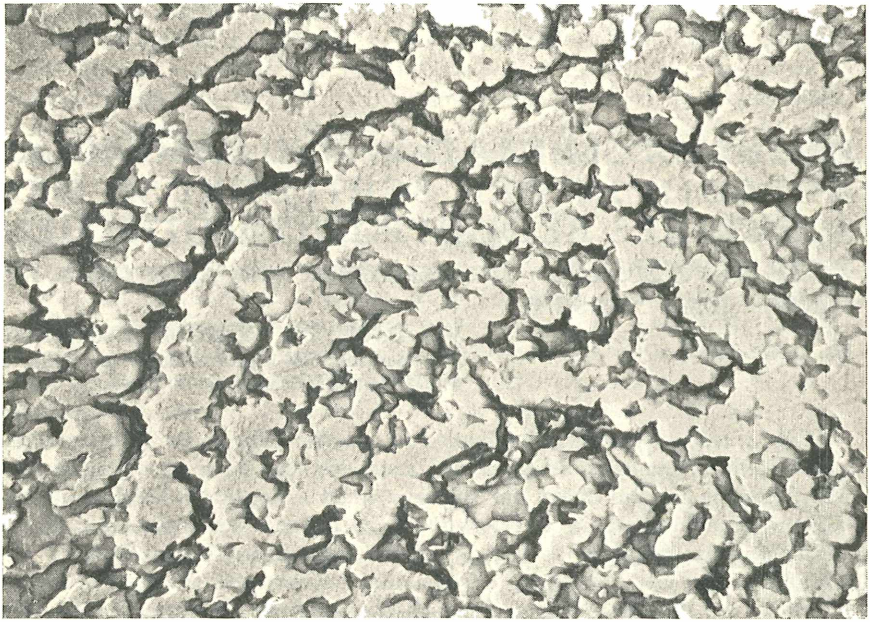


Fig. 15 and 16. Particles of carbonyl iron powder. 10.000 : 1.

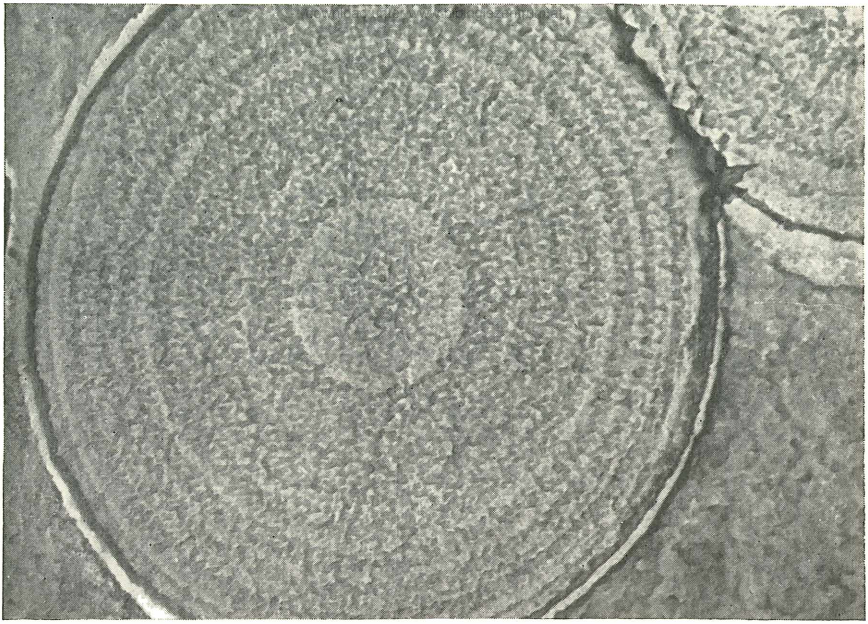


Fig. 17 Particles of carbonyl iron powder. 15.000:1.

EPITHELDEGENERATION UND BASOPHILES KOLLOID IN DER SCHILDDRÜSE DES SCHWEINES BEIM HYPOTHYREOTISCHEN ZWERGWUCHS / EIN VERGLEICH MIT ÄHNLICHEN BEFUNDEN BEIM MENSCHEN

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Mit 3 Abbildungen

Wohl infolge der schlechten Ernährung in den Jahren nach Kriegsende, andererseits wegen des besonderen Umstandes, daß in diesen Jahren nur wenig Regen fiel und daher die Pflanzen nicht Gelegenheit hatten, die benötigten Mineralstoffe in gelöster Form im Boden vorzufinden, also selbst an ihnen verarmten, traten seit 1945 bei den Haustieren in enzootischer Verbreitung Fälle von Myxödem, Struma und thyreogenem Zwergwuchs auf. Hierüber haben aus dem pathologisch-anatomischen Institut der Wiener