APPLICATION OF THE SCANNING ELECTRON MICROSCOPE IN THE EXAMINATION OF POWDERS

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Among the most important physical properties of powders, determining their potential usefulness in practical application, are the shape, size, and surface condition of their particles. The present work was undertaken with the aim of studying these characteristics by optical and transmission electron microscopy and also by scanning electron microscopy. An advantage of scanning electron microscopy is that it gives a much (some 300 times) greater depth of focus compared with the optical method, making it possible to reveal very fully the topography of particle surfaces. This considerable depth of focus, coupled with the wide range of magnifications that can be employed (from 50 to 200,000 diameters), enables selected portions of particles to be examined in detail without any additional adjustments. Furthermore, with the scanning electron microscope the specimen requires no special preparation, which means that powders can be studied undeformed and uncontaminated by some preliminary treatment. Finally, few difficulties are experienced in the interpretation of data yielded by this method of examination [1-3].

In the present work, a study was made of aluminum powders of 1-5 and 15-20 μ particle sizes, coated with thin Cu and Fe films. Films of metals and inorganic compounds applied to particles enable the quality of powders to be varied within wide limits [4].

For the optical investigation of the shape, size, and surface condition of particles, use was made of an MIM-7 microscope. Figure 1 shows a copper-coated aluminum powder of 15-20 μ particle size. To obtain these micrographs, particles were placed on a mirror surface covered with an extremely fine film of inorganic oil. It is evident that, for the powder fraction under investigation, optical examination provides only meagre information concerning the shape and size of particles, and is quite unsuitable for assessing their surface condition.

With this method, immersion cannot be employed because of the possibility of particles moving about in the relatively thick layer of immersion oil. In view of this, for examination at high magnifications powder specimens were prepared, using the following procedure: 2-3 mg of dispersed aluminum was mixed with 1 cm³ of dry, powdered Protacryl plastic and poured into a ring, which was then filled up with a solvent. After solidification, the specimens were polished with a suspension of GOI paste in distilled kerosene. It will be seen from Fig. 2, however, that, even at a magnification of 1200 diameters, little additional information about powder particles can be obtained by this method. At the same time, mechanical polishing severely distorts their surfaces.

Optical microscopy can be more useful in examinations of the particle shapes of coarser powders (100-140 μ). However, micrographs of such powders (Fig. 3) may lead to erroneous conclusions regarding the surface topography of their particles. Figure 3a illustrates some irregular-shaped particles with very rough edges. Increasing the magnification fails to provide any new information concerning their surface condition (Fig. 3b and c).
Fig. 1. Dispersed coated with Cu, of 15-20 μ particle size. Magnification: a) x200; b) x480.

Fig. 2. Transverse section of dispersed Al of 15-20 μ particle size. Magnification x1200.

Fig. 3. Dispersed Al coated with Cu, of 100-140 μ particle size. Magnification: a) x200; b) x340; c) x480

The same powders were then examined by the method of transmission electron microscopy. The micrograph illustrated in Fig. 4 was obtained with the aid of a carbon replica into which particles had been incorporated by the technique proposed in [5]. The micrograph shows particles of regular, spherical shape, having smooth surfaces. However, the fact that the specimen must be specially prepared complicates investigation by this method. It will be seen that the character of particle surfaces depicted in Fig. 4 differs from that shown in Figs. 1-3. The reasons for this difference become apparent when the powder is examined with the aid of the scanning electron microscope.

In our work, examinations of powders were made at the Institute of Semiconductor Physics, Siberian Division of the Academy of Sciences of the USSR, using a Cambridge Stereoscan scanning electron microscope. The magnifications employed ranged from 500 to 19,000 diameters. Figure 5 shows micrographs of copper-coated aluminum particles. The particles were 15-20 μ in size, the area scanned, at the minimum magnification of 500 diameters, being 4 \cdot 10^4 μ² (Fig. 5a). In the field of vision can be observed an appreciable number of particles, mainly of spherical shape. Some of the larger particles exhibit fine, bright spots, which, as can be seen from Fig. 5b and c, are much smaller spherical particles adhering to the large ones. Thus, the apparent surface roughness illustrated in Fig. 3 may be attributed to the presence of these fine particles.

Figure 6 shows micrographs of iron-coated dispersed aluminum particles 1-5 μ in size. The particles of this fraction, too, have spherical or near-spherical shapes. An examination of micrographs obtained at increasing magnifications revealed that the powders investigated were composed of particles