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Fine structure of electron-diffraction spots due to particle shape and to the method of shadow-casting.

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1. The multiple fine structure of electron-diffraction rings due to the refraction effect of electrons at surfaces of crystalline particles, which has been noted on smoke particles, such as magnesium oxide, having a clean-cut cubic habit, furnishes us with a method of determining the shape of microcrystals by electron diffraction. We will give two examples of this method:

(i) Manganous oxide formed on a manganese–copper alloy by the selective oxidation of manganese, and barium oxide formed by the decomposition of barium carbonate at the primary stage of the formation process of oxide cathodes, show a characteristic fine structure of diffraction rings similar to that of magnesium-oxide smoke, indicating a cubic habit of the particles.

(ii) As has been formerly reported, a characteristic fine structure of diffraction spots due to smoke particles of zinc oxide can be explained well when we assume the needle-like particles to be hexagonal prisms bounded by ((10.0)) planes (Rees & Spink, 1950; Honjo, Miyake & Mihama, 1950). Similar fine structures of diffraction spots are also found for unoxidized metallic particles of magnesium smoke and seem to indicate the same needle-like crystalline habit.

2. As we have previously reported, the smoke particles of magnesium oxide deposited on a flat surface, such as the cleavage face of a crystal, are disposed as dice on a flat table, so that they assume a fibrous orientation with [001] perpendicular to the underlying surface; the diffraction spots in the fibrous pattern due to electrons incident at a grazing angle to the substrate are then generally doublets split in the horizontal direction (Fig. 1 (*a*)), in accordance with the refraction effect at surfaces of crystallites (Miyake & Honjo, 1950). When such a smoke sample is *shadow-casted* by evaporating chromium metal in a direction of incidence grazing the substrate and nearly perpendicular to the electron beam, the member of the doublet deviated in the opposite direction to the source of metallic vapour disappears, apparently as the result of the fact that the deposited metal screens a pair of surfaces of the smoke particles, facing the metallic vapour. The effect can be seen most distinctly on (111) and (113) reflexions (Fig. 1 (*b*)), in which only the particles having a particular azimuth in relation to the electron beam are capable of participating. The shadow-casting does not otherwise affect the diffraction pattern except to produce a slight enhancement of the background.

The (004) and (006) reflexions located on the central line in the fibrous pattern also split into well-defined doublets (Fig. 2 (*a*)) when the incidence of electrons is such that the mirror reflexion of the substrate surface approaches their respective positions. This is also explained by the refraction effect, since the expected extinction of the component spot by the shadow-casting was observed. The sharpness of the deviated spots, in spite of the fact that the particles taking part in the reflexions concerned may take any arbitrary azimuth around the fibre axis [001], can readily be explained by a simple calculation which shows that most of the deviated diffractions concentrate in a narrow range close to the positions corresponding to the minimum deviations.

The (002) reflexion, on the other hand, shows a quartet structure under similar conditions (Fig. 2 (*b*)). It was found that the shadow-casting extinguishes the two of the quartet spots opposite to the source of metallic vapour (Fig. 2 (*c*)). Fig. 2 (*d*) shows the intermediate stage of the extinction. From this observation it is known that the quartet structure may be caused by the double refraction which is expected by the dynamical theory of electron diffraction (Sturkey, 1948). In fact, theoretical estimation of the effective inner potentials shows a reasonable agreement with the observed separations and, at the same time, indicates that both the (004) and (006) doublets mentioned above should be regarded as unresolved quartets.

A number of multiplet spots due to individual smoke particles, distinctly showing the double refraction, is also observed in the general positions on the Debye–Scherrer rings of our smoke sample (Fig. 3), the details of which effect, however, seem to require a more refined consideration than that presented by Sturkey. A fuller discussion on this point will be given at a later date.

3. Another example of the application of the shadow-casting method is presented in Fig. 4, the diffraction pattern of the smoke sample of zinc oxide shadow-casted with the direction of incidence perpendicular to the electron beam as indicated by the arrow. It may be seen, for example on the (11.0) ring, that the spots in the vicinities of the two ends of the diameter of the ring parallel to the arrow have commonly lost their components opposite to the source of metallic vapour, whereas there is no change in the spots near the ends of the diameter perpendicular to the arrow. This is quite in accordance with the interpretation for the fine structure in terms of the particular shape of the smoke particles as mentioned above, taking into account the refraction effect as well as the effect of crystal shape.

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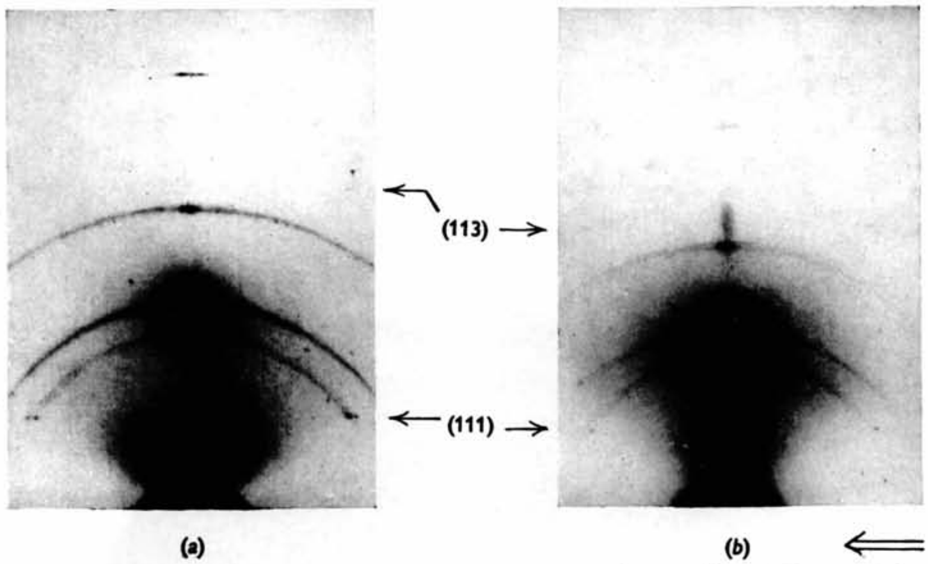

Fig. 1. (*a*) Fibrous pattern due to smoke particles of magnesium oxide. (*b*) Pattern of the smoke particles shadow-casted with metallic vapour incident in the direction indicated by the arrow.

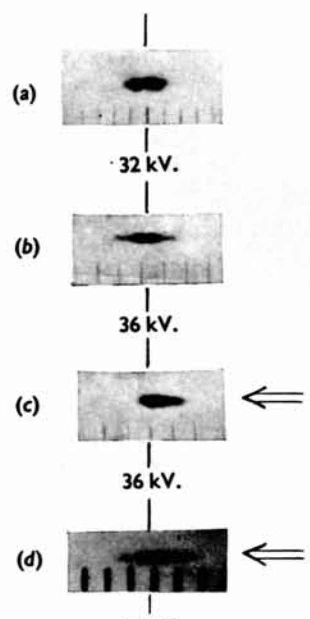

Fig. 2.

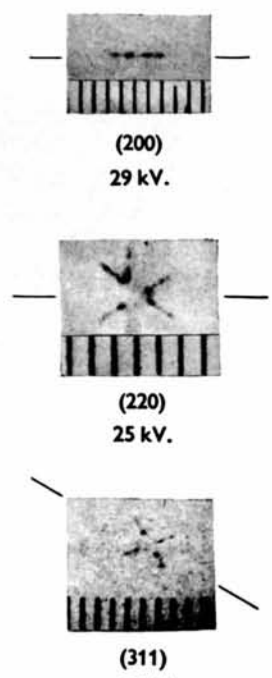

Fig. 3.

Fig. 2. Multiple structures of (00*l*) reflexions due to fibrous smoke sample of magnesium oxide. (*a*) (004) doublet. (*b*) (002) quartet. (*c*) Extinction of two component spots of the quartet by shadow casting. (*d*) Intermediate stage of the extinction. (One division of the scale corresponds to 0·1 mm. on the original plate taken by an electron-diffraction camera of length 15 cm. with the accelerating voltages shown.)

Fig. 3. Multiple structures due to individual smoke particles of magnesium oxide showing double refraction. (One division of the scale corresponds to 0·1 mm. on the original plate taken by an electron-diffraction camera of length 15 cm. with the accelerating voltages shown.)

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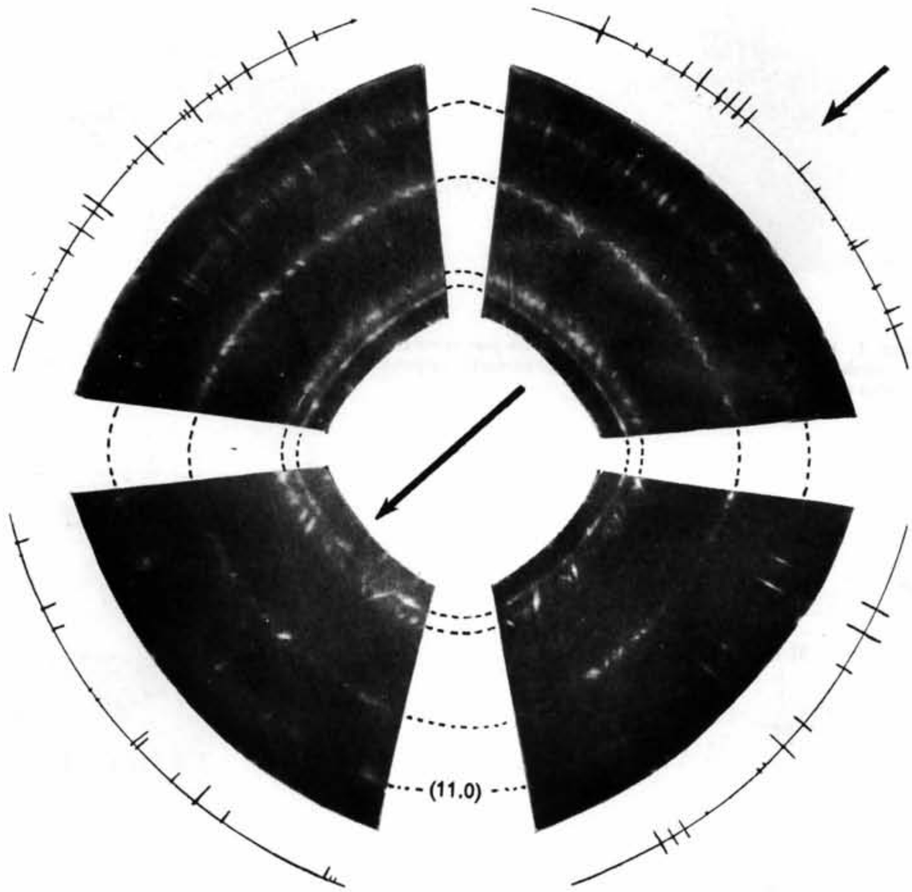

Fig. 4. Diffraction pattern due to smoke sample of zinc oxide shadow-casted with metallic vapour incident in the direction indicated by the arrow. The effect of shadowing on the (11.0) ring is shown schematically.