

latter one the reaction is not so far advanced as in the former. The micrograph of Fig. 1 was obtained from a specimen aged for $1\frac{1}{2}$ hr. at 350°C so that it is in approximately the same condition as the crystal of Fig. 8 which is not yet in the final state of equilibrium.

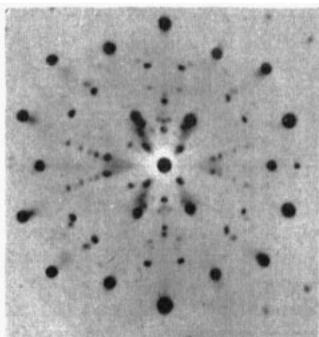


Fig. 8. Another crystal aged 1 hr. at 350°C : condition as in Fig. 1, orientation as in Fig. 7

The silver-aluminium alloys go through a similar sequence: formation of plates—intermediate phase—final phase. In this case, however, the plates of silver-rich material form on the (111) planes of the matrix and the intermediate phase is a close-packed hexagon, the basal plane of which fits accurately on to the (111) plane of aluminium. The axial ratio of this new phase is 1.61, nearly, but not quite, the value $\sqrt{8/3}$ for the packing of spheres. There is therefore a slight misfit in the direction at right angles to the plane of union of the two phases,

which will strain the lattice of the matrix. With rise of temperature and prolongation of treatment this unstable phase transforms into the final precipitate which also has a close-packed hexagonal structure, but with a parameter which does not fit the aluminium lattice at all accurately.

The general course of the two reactions outlined above is fairly well known, but there are details as to the time and temperature required to produce a given state that still require investigation. The use of powerful sources of monochromatic radiation is necessary to exploit the experimental method fully and should yield further information about reactions of this type.

The work described above has been carried out as part of the research programme of the National Physical Laboratory, and this paper is published by permission of the Director of the Laboratory.

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PARTICLE SIZE MEASUREMENT BY THE X-RAY METHOD. BY F. W. JONES,

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ABSTRACT. The X-ray method of particle size determination is briefly reviewed. A short summary is given of the theoretical work and the more important experimental methods. The accuracy and limitations of the method are discussed.

INTRODUCTION

THIS method of particle size measurement is applicable to polycrystalline specimens where the crystal size is in the range from about 10^{-5} to 10^{-7} cm. The experimental arrangement adopted is generally the usual Powder Method, where a polycrystalline specimen of cylindrical form is mounted at the centre of a cylindrical camera round which the film is wrapped. If the particle size of the crystals is in the measurable range, the X-ray lines are broadened. The breadths of the lines may be measured by means of a microphotometer. Two different methods of attack are then available for obtaining the particle size from the measured breadths:

(1) The particle size may be obtained directly from curves giving the line breadth as a function of the particle size. Such curves have been calculated by Brill and Pelzer^(1,2,3) and by Kochendörfer⁽⁴⁾ for various experimental arrangements.

(2) The measured breadths may be corrected by an empirical method for the broadening caused by the experimental conditions, so as to obtain the pure diffraction breadth (i.e. the breadth which would be obtained using the ideal experimental conditions of a point specimen and parallel monochromatic radiation), from which the particle size may be found⁽⁵⁾.

The particle size may be determined independently from several lines on the film. Only when the crystals are approximately cubic or spherical in shape are the results likely to agree. Where they differ, it may be possible to obtain some information about the particle shape^(5, 6).

THEORETICAL

The fundamental theoretical problem is the calculation of the pure diffraction breadth of the X-ray lines in terms of the size and shape of the crystals. All the theoretical workers agree upon a formula of the type

$$B = \frac{K\lambda}{\epsilon \cos \frac{1}{2}\chi},$$

where B is the angular breadth of the line (defined below), λ the wave-length of the X-radiation and χ is the angle between the incident and diffracted rays. In the case of cubic crystals of cubic lattice type ϵ is the edge length of the crystals, but in general ϵ is a function of the size and shape of the crystals and the Miller indices h, k, l . Different values of the constant K have been given. It is approximately equal to unity. Two different definitions of breadth have been used, the 'half-value breadth' which is the breadth at half the maximum intensity, and the 'integral breadth', defined as

$$B = \int \frac{I d\chi}{I_{\max}},$$

where I is the intensity at the angle χ , the integration to extend over the X-ray line. As these two definitions

will not in general give the same result, it is necessary to ascertain which has been used in comparing the results obtained by various theoretical workers. The integral breadth is usually more convenient for use where the breadth of the line is influenced by the geometry of the apparatus.

The theoretical work on the problem is summarized below:

Scherrer(7). Formula for a cubic crystal of cubic lattice type, the edges of the cubic crystal being parallel to the crystal axes.

Seljakow(8). Generalization of Scherrer's result to non-cubic crystals. He assumed, however, that the external form of the crystal was similar to that of the unit cell.

Bragg(9). A very simple calculation of the line breadth in terms of the thickness of the crystal normal to the reflecting planes.

Laue(10). The most general theoretical attack on the problem. The paper consists of two parts:

(a) The calculation of the line breadths in terms of the Miller indices and the edge lengths $N_1 a_1$, $N_2 a_2$, $N_3 a_3$ of the parallelepiped crystal, which are assumed to be parallel to the axes of the unit cell of which a_1 , a_2 , a_3 are the parameters.

(b) The application of the formula derived in (a) to the experimental case of a perfectly transparent specimen illuminated by purely divergent radiation.

The first part of the paper has provided the background for most of the subsequent work, but it is somewhat difficult, requiring a knowledge of the reciprocal lattice, and it contains two arithmetical errors (5). In his treatment Laue made approximations which affect the value of the constant in his final formula. Later workers have been concerned with the more exact evaluation of this constant(5, 11, 12) and with the effects of the shape of the particles(13, 14, 15, 16).

EXPERIMENTAL

Brill and Pelzer have given methods by which allowance is made for the line broadening caused by the experimental conditions (which give the X-ray a definite width, even when the diffraction broadening is negligible). The most important are their hollow sample method (1, 2) and their total absorption method (3). Their results are published in the form of curves which give the particle size as a function of the line breadth at different angles.

Kochendörfer(4) has made similar calculations for a specimen in the form of a flat plate.

According to the method of Jones(5) a powder of large crystal size ($> 10^{-4}$ cm. edge length) is mixed with the powder of unknown crystal size. The breadths of the lines due to the standardizing substance having been measured, the breadths of the lines due to the powder of unknown size may be corrected for the experimental conditions.

Jones's method is somewhat more laborious than those of Brill and Pelzer, but it involves less assumptions and is more elastic as regards the experimental arrangements.

Taylor(19) has suggested a simplification of Jones's method of obtaining the correction curve whereby the arithmetic is considerably reduced.

Brill(6) and Jones(5) have given methods by which the particle shape may be determined from measurements on lines of different Miller indices.

ACCURACY AND LIMITATIONS OF THE METHOD

Under the best conditions, and when the particle size is about 100 Å, a reproducibility of the order of ± 10 per cent may be obtained. The accuracy decreases as the particle size increases and with the usual experimental arrangements it is difficult to detect the line broadening when the particle size is 1000 Å. As the particle size decreases the accuracy also decreases somewhat as excessive broadening weakens the X-ray lines. The absolute accuracy is very probably considerably lower than the reproducibility. Little has been done in the way of checking the results by other methods, but attempts have been made by Cameron(17) and Holley and Bernstein(18). It is not to be expected that the crystals are all the same size and the observed mean size is likely to differ from the true mean size(5).

The presence of lattice strain will introduce uncertainty into the results, but it is probably safe to assume that measurements made under such conditions will give a minimum figure for the particle size.

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The subject has been reviewed fairly recently by Cameron and Patterson (American Society for Testing Materials—*Symposium on Radiography and X-Ray Diffraction*, 1936), who give a comprehensive bibliography.

INDUSTRIAL RADIOLOGY GROUP

At a meeting held at Harrow on 20th May last, and attended by some seventy persons from all over the country, an Industrial Radiology Group was formed under the auspices of the Institute of Physics. Its object is to provide a medium for the exchange of information on any matter relating to the application of X-rays or gamma-rays to industrial problems. It is hoped that the Group may, in due course, issue a Report summarizing many of the essential practical points of im-

portance to users of X-ray plant. A committee has been appointed representing those employing X-rays in industry and the manufacturers of industrial X-ray apparatus and films. Membership of the Group is open to all interested, whether at present members of the Institute of Physics or not. Further particulars may be obtained from the Honorary Secretary of the Group, Dr L. Mullins, of the Research Laboratory, Kodak Ltd., Harrow, Middlesex.