allowed to proceed for only a short time (about 6 min.). The Fe_2S_3 may be unstable when the sulphide film thickness becomes several thousand Ångströms or more, since further growth (reaction for 30–45 min.) leads to the formation of nearly pure normal FeS. Epitaxial misfit on the substrate may have affected the stability.

As is to be expected, the action of hydrogen sulphide is not inhibited by an oxide film at these temperatures, probably owing to a reaction of the type

$$2\,Fe_3O_4+9\,H_2S \rightleftarrows 3\,Fe_2S_3+8\,H_2O+H_2$$

being driven over to the right by the high concentration of hydrogen sulphide.

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A Note on the Measurement of Lattice Spacings from Unannealed Powders or Filings

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A procedure is described for measuring the lattice parameters of cold-worked powder specimens which may, or may not, contain deformation stacking faults.

1. Introduction

The measurement of lattice spacings of annealed powders is now an accepted technique, and an accuracy of the order 1 part in 30,000 to 1 part in 100,000 can be obtained by standard extrapolation methods. In the study of some alloys of high melting point, the measurement of lattice spacings from unnealed powders or filings may be valuable. It is, for example, difficult to anneal and quench filings from temperatures above 2000° C., although lumps of alloy may be quenched satisfactorily, and, from these, filings may be prepared and examined in the cold-worked state. Where the etching of alloys is difficult the determination of phase-boundaries by lattice-spacing measurements is attractive, and should be possible if accurate determinations can be made on cold worked material. The present note describes a method by means of which lattice spacings can be measured to an accuracy of the order 1 part in 3,000 to 1 part in 10,000, using un-annealed powders or filings.

2. General method

Anantharaman & Christian (1953) have described an analytical method based on Rachinger's graphical procedure by means of which the line profile of a diffuse Kα line may be resolved into its constituent $K\alpha_1$ and $K\alpha_2$ profiles. The method assumes that the lines are symmetrical about their peaks, and that the intensity ratio of $\alpha_1:\alpha_2$ is 2:1. Subject to this assumption, overlapping doublets which have been broadened symmetrically by cold work may be resolved into their constituent α_1 and α_2 profiles. Variation in background intensity must be allowed for.

In the present work, the line profiles were measured by standard methods on a Philips Geiger-counter X-ray diffraction unit, using flat powder specimens. It was sometimes necessary to apply a dead-time correction for counter losses.

3. Body-centred cubic structures

For these structures the method is straightforward, because stacking faults are not produced by cold-working. All lines are broadened symmetrically, and the peak positions are not affected. It is, thus, necessary only to analyse the profiles of three or more lines, and to follow the normal extrapolation procedure. In order to test the method, experiments were made on pure iron, and on iron–chromium alloys whose lattice spacings had been measured in a 19 cm. Unicam camera by Sutton & Hume Rothery (1955). The results are shown in Table 1, from which it will be seen that the greatest difference between the lattice spacings determined by the two methods is 0·0005 kX.

Table 1. *Body-centred cubic structures*

Material	a_0 (kX.), present method (unannealed)	a_0 (kX.), normal procedure (Sutton & Hume-Rothery)
Pure Fe	2·860(1)	2·8604
	2·860(1)	
Fe–5·37 at.% Cr	2·863(6)	2·8633
	2·863(8)	
Fe–3·41 at.% Cr	2·862(5)	2·8622
Fe–2·26 at.% Cr	2·861(9)	2·8616
	2·861(7)	

4. Close-packed hexagonal structures

These materials may contain both growth and deformation stacking faults. Expressions for the diffracted intensity have been given by Wilson (1949, growth faults), Christian (1954, deformation faults) and Gevers (1954, both kinds co-existing). Extrinsic faults (Frank & Nicholas, 1953) have not been treated, and are not considered here.

If we use the capital letters H, K, L to denote the Miller indices of planes in the close-packed hexagonal lattice, the effect of both kinds of fault is to broaden all reflections with $H-K = 3M\pm1$ and L odd or even *symmetrically* in the L direction in reciprocal space. Reflections with $H-K = 3M$ and L even are unbroadened by faults. When dealing with diffraction by powders, the lines with $L = 0$ are virtually unbroadened by faults and so it is possible to arrange, by suitable choice of radiation, that there shall be lines not broadened by faults at Bragg angles $> 45°$.

The procedure described in § 2 is therefore applicable to materials with hexagonal structures.

5. Face-centred cubic structures

Growth faults in these structures produce asymmetrical line broadening and peak shifts. Their occurrence is unlikely in deformed powders and they are neglected in the present work. Deformation faults produce symmetrical line broadening and peak shifts (Paterson, 1952), consequently the simple extrapolation method cannot be used. This difficulty may be overcome by the following treatment:

For faults on the (111) planes the shift in peak of an $\{hkl\}$ diffraction line in degrees is given by

$$\delta\theta = \frac{\pm\tan\theta\cos^2\varphi\,270|/3\,.\,\alpha}{2\pi|h+k+l|}\,, \qquad (1)$$

where φ is the angle between the reflecting normal and the planes containing deformation faults, and α is a stacking-fault parameter.

The mean value of $\cos^2\varphi$ has to be taken for the various planes (hkl), $(\bar{h}kl)$, $(h\bar{k}l)$ etc. Of the $\{111\}$ family the (111) and $(\bar{1}\bar{1}\bar{1})$ components are not shifted, whilst those of type $(\bar{1}11)$ move to higher Bragg angles. For the $\{200\}$ family, all components move to lower Bragg angles. It is assumed that the effect of faults on other $\{111\}$ planes is additive.

Denote the extrapolated true value of the lattice parameter (i.e. that for a strain-free material) by a_0 and the values obtained from the cold-worked α_1 peak positions by a', a'', a''', ... for the various lines measured. (The procedure adopted normally is to plot the various values of the lattice parameter for these lines in the annealed material a_1, a_2, a_3, ... against the extrapolation function, $f(\theta)$, which has values x_1, x_2, x_3, ... for the lines chosen. A straight line is obtained and extrapolated to $f(\theta) = 0$.) Let the slope of this line be m; then the general equation is

$$a_i = mx_i + a_0, \qquad (i = 1, 2, 3, \ldots)\,. \qquad (2)$$

The error in a determination of a_i caused by an error $\delta\theta$ in the Bragg angle is $a_i\cot\theta\,.\,\delta\theta\,.\,2\pi/360$, where θ is in degrees.

$\delta\theta$ in this case is the shift in peak position due to faults. Combining this with equation (1), the error in a determination of a_i is given by

$$\frac{a_i\cos^2\varphi 3|/3\,.\,\alpha}{4|h+k+l|} = p_i(\alpha)\,. \qquad (3)$$

For the various lines,

$$\left.\begin{aligned} a' &+p_1(\alpha) = a_1\,,\\ a'' &+p_2(\alpha) = a_2\,,\\ a''' &+p_3(\alpha) = a_3\,, \end{aligned}\right\} \qquad (4)$$

which, together with (2), gives

$$\left.\begin{aligned} a' &+p_1(\alpha)-mx_1-a_0 = 0\,,\\ a'' &+p_2(\alpha)-mx_2-a_0 = 0\,,\\ a''' &+p_3(\alpha)-mx_3-a_0 = 0\,. \end{aligned}\right\} \qquad (5)$$

These are three unknowns, m, α and a_0 so *at least* three lines must be measured. By eliminating m and α from equations (5) the equation

$$a_0\left\{\frac{x_2-x_1}{p_2x_1-p_1x_2}-\frac{x_3-x_1}{p_3x_1-p_1x_3}\right\} + \frac{a''x_1-a'x_2}{p_2x_1-p_1x_2}-\frac{a'''x_1-a'x_3}{p_3x_1-p_1x_3} = 0 \qquad (6)$$

is obtained, giving a_0 in terms of the known quantities. From a similar equation α can be obtained.

To test the method, it has been applied to specimens which it is possible to anneal, and which contain large amounts of faults. More than three lines were used, and so the results from equation (6) could be checked. The usual method of measuring stacking faults is to measure the changes in separation of adjacent pairs of lines on annealing the cold-worked filings.

Table 2 compares the values of a_0 and α obtained by the present method with those obtained by the normal procedures. The cold-worked lines measured were low-angle lines, since the primary object of the work was to measure stacking faults. For the annealed materials, high-angle lines were also measured to obtain the lattice parameter. The differences between the a_0 values are within $\pm0{\cdot}001$ kX. for $\alpha\sim0{\cdot}02$ and

Table 2. *Face-centred cubic structures*

Material	Present method		Normal procedure	
	α	a_0 (kX.)	α	a_0 (kX.)
Co–Ni (31 wt. % Ni)	0·022	3·529(0)	0·022	3·529(5)
Co–Fe (7·9 wt. % Fe)	0·023	3·544(3)	0·023	3·544(5)
Cu–Al (4 wt. % Al)	0·016	3·631(2)	0·013	3·630(7)
Cu–Al (6 wt. % Al)	0·031	3·644(7)	0·031	3·642(2)
Pure silver	—	4·077(7)	—	4·0775*

* Accepted value.

become larger for increasing α, presumably because the accuracy of the method of locating the α_1 peak positions of the lines decreases with increasing α. Table 2 also gives a result of a_0 for pure silver, where it was found that the a' values lay on a good straight line, from which it was concluded that the faulting was negligible. The agreement between the value as found and the accepted value for pure silver is good.

6. Conclusions

For materials which it is difficult to strain-anneal satisfactorily, and where difficulties of grain size preclude the use of solid rod specimens in conventional powder cameras, the analytical method may be applied to deformed powders using a Geiger-counter diffractometer. Even in the unfavourable case of faulted face-centred cubic structures, lattice spacings may be obtained to $\pm 0{\cdot}001$ kX. for values of α up to 0·02, and for body-centred cubic structures the accuracy is considerably greater.

For more complex structures, complications from deformation faulting are improbable and, as for the close-packed hexagonal structures referred to above, the simple method should be applicable, provided that sufficient lines can be obtained which are not overlapped by other reflexions.

The time involved is of the order of three times that of the conventional method, and if allowance is made for the time of annealing, which in certain cases may not even then be satisfactory, it is clear that the above procedure is practicable.

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Electron-Optical Observations with Crystals of Antigorite*

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Electron-optical fringes are obtained from an antigorite (Yu Yen Stone) which agree in spacing and direction with the superlattice parameter $a = 100 \pm 10$ Å determined by electron diffraction. The nature of the superlattice is discussed.

In the course of a combined electron-diffraction (E.D.) and electron-microscope (E.M.) study of serpentine minerals (Zussman, Brindley & Comer, 1957, hereinafter labelled Z.B.C.), an unusual phenomenon was observed with a variety of antigorite from Manchuria called 'Yu Yen Stone', (U.S. National Museum, No. 94356). This material is massive in the hand specimen but finely ground powder yields single platy crystals suitable for E.D. and E.M. study. The phenomenon is illustrated in Fig. 1, which shows high-magnification

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