In this structure each Si is surrounded by 12 Mo at 2·73 A. Each Mo has 2 Mo at 2·44 A., 4 Si at 2·73 A. and 8 Mo at 2·99 A.

Table 1. *Diffraction data for* Mo_3Si

hkl	*d/n**	Intensity: Visual†	Intensity: Spectrometer	Intensity: Calc.
110	3·458	*m*	22	36
200	2·445	*m* –	21	25
210	2·187	*vs*	122	183
211	1·996	*s* –	55	58
220	1·729	*w*	5	3
310	1·546	*w* +	6	7
222	1·412	*m*	22	25
320	1·356	*m* +	39	35
321	1·307	*m* +	26	26
400	1·222	*m*	21	16
411, 330	1·153	*w*	3	4
420	1·093	*m* –	—	7
421	1·067	*s*	—	33
332	1·043	*m* –	—	7
422	0·9982	*w* –	—	2
510, 431	0·9590	*w* +	—	6
520, 432	0·9080	*s* +	—	43
521	0·8928	*m*	—	13
440	0·8644	*m* +	—	22
530, 433	0·8386	*w*	—	5
600, 442	0·8150	*m*	—	12
610	0·8039	*s* –	—	27
611, 532	0·7933	*s*	—	39

* $a=4·890$; Cu $K\alpha_1=1·54050$ A.

† *vs* = very strong; *s* = strong; *m* = medium; *w* = weak.

Thus molybdenum silicide is isomorphous with Cr_3Si (Borén, 1933; *Strukturbericht*, 1937*c*) and V_3Si (Wallbaum, 1939; *Strukturbericht*, 1939*a*, *b*). The structure is that of β-wolfram, which has 8 W per unit cell, located in both the molybdenum and the silicon positions (Hartman, Ebert & Bretschneider, 1931; *Strukturbericht*, 1937*a*, *b*). The compound UH_3 is similar, with 8 U in the same structure and with hydrogen in certain interstitial positions (Rundle, 1947).

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A New Weissenberg Technique Using a Double Slit

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The paper describes a new method of recording the *n*th and zero layers of the reciprocal lattice simultaneously by means of a Weissenberg goniometer with two screen slits. The new method combines the principal advantages of the oscillation and equi-inclination techniques for measuring X-ray intensities by allowing the intensities of both layers to be recorded on the same relative scale. The anti-equi-inclination position is used for the zero layer, and the application of the Lorentz factor and absorption corrections for this position is discussed.

1. Introduction

Because of its great simplicity of interpretation the Weissenberg moving-film technique is now being used increasingly in place of the more cumbersome oscillation technique for X-ray crystallographic work in general and the photographic determination of intensities in particular. In this connexion a series of new problems present themselves, some of which will be dealt with in this paper.

In order to obtain a set of intensities from planes of the type (*hkl*), it is necessary to correlate photographs of different layers as well as those taken about different axes. The method used for the correlation of oscillation photographs (Cox & Shaw, 1930) is not applicable to Weissenberg photographs where different layers are not recorded simultaneously and can therefore be brought to the same scale only by comparison with other zones. Thus, for instance, the factor *K* between the (*h*0*l*) and (*h*2*l*) reflexions recorded about the [*b*] axis is found by correlation via, say, the (00*l*), (02*l*) of

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the $(0kl)$ set about $[a]$. For this purpose the factors K_{00} between $(00l)_b$ and $(00l)_a$, and K_{02} between $(02l)_b$ and $(02l)_a$ are calculated from the ratios of corresponding intensities; then $K=K_{00}/K_{02}$. Now, individual estimates of intensities are subject to relatively large errors, and it is therefore desirable to calculate the correlation factor from an average of as many reflexions as possible. In the method outlined only the few intensities of the type $(00l)$ and $(02l)$ are available so that the error in K, which contains the cumulative errors in K_{00} and K_{02}, may be considerable. Moreover, the sets of intensities used are taken about different axes, which in many cases means that different crystals have been employed; these may be of different shape, and give spots of different size, subject to different extinction and absorption errors which can never be completely eliminated.

2. The new method

To overcome these difficulties a new technique has been devised whereby any selected layer can be recorded simultaneously with the zero layer, and at the same angle of incidence, i.e. on the same relative intensity scale without any further need for correlation. The anti-equi-inclination position (Buerger, 1942, p. 301) is utilized for the zero layer, as can be most easily understood from Fig. 1.

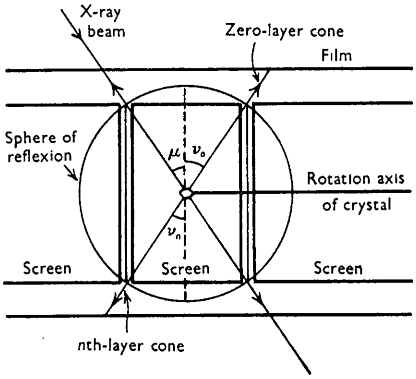

Fig. 1. Diagram showing the position of the slits relative to the sphere of reflexion and to the nth and zero layers of the reciprocal lattice.

The incident X-ray beam makes an angle μ with the normal to the rotation axis, and the cone of reflexion for the nth layer makes an angle ν_n with this plane such that $\nu_n=\mu$, the condition for equi-inclination. The zero layer will go through the point where the X-ray beam emerges from the sphere of reflexion, and its cone will therefore make an angle $\nu_0=\mu$ with the normal to the rotation axis. The zero- and nth-layer cones therefore make the same angle with the rotation axis and are allowed to reach the moving film through two slits in a stationary screen instead of through the single slit for the nth layer only as used in the ordinary equi-inclination Weissenberg method. If the radius of the screen is r, the slit centres will have to be at $r \tan \mu$ on either side of the crystal, and if the slit width is s, the centre portion of the screen will be of width $2r \tan \mu - s$; it is therefore merely necessary to insert a ring of that width between the screens normally provided with the instruments.

3. Interpretation of photographs

The photograph produced by this method (Fig. 2) is, of course, more complex to analyse than the ordinary single-slit photograph, since it virtually consists of two such photographs superimposed with a lateral shift of $2R \tan \mu$, where R is the effective film radius. But provided the cell dimensions are not too large, this does not present any great difficulties, especially since both sets of reflexions can be indexed using the ordinary Weissenberg chart for equi-inclination photographs. To simplify the task still further, one of the slits can be opened to one half of the film only, or the constituent photographs can be taken separately to facilitate indexing, although in the case investigated (cell dimensions about $7\times8\times10$ A.; Cu $K\alpha$ radiation) this was found quite unnecessary. It is useful, however, to prepare a glass chart, spotted in two colours to correspond to the respective positions of each set. Indexing then presents little more difficulty than with the ordinary Weissenberg photographs, and one chart will usually serve for all photographs around one axis,

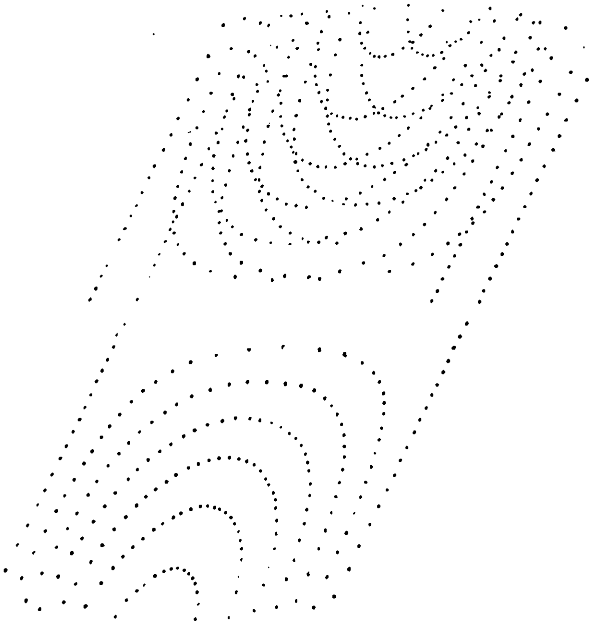

Fig. 2. Spot chart showing the pattern obtained on a double-slit photograph. Complete layers of orthogonal mesh $0{\cdot}1\times0{\cdot}3$ reciprocal-lattice units $0{\cdot}4$ reciprocal-lattice units apart are shown. In an actual photograph some of the spots will be absent for space-group reasons, or because they are too weak to reflect. Only a single layer is contained in the lower half of the figure.

since the distortion of the ξ-scale for different inclinations is small ($\cos \nu$).

4. Correction of intensities

The intensities are subject to Lorentz, polarization and absorption corrections, and these will be discussed in turn.

The Lorentz factor can be expressed in the general form $1/L = \cos \mu \cos \nu \sin \Upsilon$, where Υ is the reflexion azimuth (Buerger, 1940); this reduces to $1/L = \cos^2 \mu \sin \Upsilon$ in both equi- and anti-equi-inclination. $\cos^2 \mu$ is constant for each photograph, so that $\sin \Upsilon$ is the only required correction; this is best read off for each spot from a chart with parallel contours of value $\sin \Upsilon$. For the standard Weissenberg camera (film-radius 57·3 mm.) $\Upsilon = 2x$, where x is the distance in millimetres from the zero line. For an orthogonal axis the Lorentz correction is equal for corresponding spots on the zero and nth layers.

The polarization factor is that normally employed, since it depends on θ alone, but special mention should be made of absorption corrections. These have been tabulated (*Internationale Tabellen*, p. 584) for the equatorial reflexions from cylindrical powder or single-crystal specimens, and apply directly to zero layers of normal-beam oscillation and Weissenberg photographs, but not to higher layers; nor can they conveniently be adapted for this purpose. For the case of equi-inclination or anti-equi-inclination, however, a simple modification allows the use of these tabulated values. Comparing the path made by the incident and reflected rays in either of these two positions with that in the equatorial case, each path $s \sec \nu$ in the former will be found to correspond to a path s in the equatorial case, where the factor $\sec \nu$ enters because both the incident and reflected beams are at angle ν with the normal section of the cylinder. Since $\sec \nu$ is constant and enters into every path over which the integration in the normal section has been carried out, it remains as a constant factor outside the integral, so that direct use can be made of the tabulated values for absorption by replacing the radius r of the cylindrical specimen by $r \sec \nu$. The angle θ of the table is then no longer the Bragg angle, but merely half the angle measured on the projection (i.e. $\frac{1}{2}\Upsilon$ in Buerger's notation).

In double-slit photographs, then, correction for absorption is easily effected on cylindrical specimens. It is, moreover, equal for corresponding spots on the nth and zero layers along an orthogonal axis.

5. General remarks

The method is obviously capable of extension to more than two slits. A third slit half-way between the other two, for instance, will record the $(\frac{1}{2}n)$th layer in the flat-cone position and could be correlated with the zero layer by the method of Cox & Shaw (1930). However, the advantage of having more than two layers recorded simultaneously is outweighed by the difficulty of indexing general inclination Weissenberg photographs, and by the increasing complexity of the photograph as the number of spots increases. Also the background fogging, already more than doubled in a double-slit photograph, would be still further augmented.

On the other hand, a double-crystal-double-slit method will allow direct comparison of the nth layer of any crystal with the zero layer of a standard crystal and will thus extend the usefulness of any double-crystal instrument.

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