determination of the integral distribution function. Further, the differences in $\langle I \rangle$ depend on less subtle principles than do the differences between the distribution functions, and are less likely to be upset by deviations from the ideal of a random distribution of a large number of atoms of approximately the same scattering factor. Heavy atoms in general positions should not be troublesome, though those in special positions may cause anomalies.

The writer's thanks are due to Dr D. Rogers, whose unpublished work on Patterson syntheses showed qualitatively that effects of the type discussed here must exist and should be sought.

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The Crystal Structure of Mo_3Si

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The crystal structure of Mo_3Si has been determined from powder-diffraction patterns. The lattice is cubic with $a = 4{\cdot}890 \pm 0{\cdot}002$ A. The structure is that of β-wolfram and the compound is isostructural with Cr_3Si and V_3Si.

An investigation of the phase diagram of the molybdenum–silicon system has been made in this laboratory by Searcy (1949) and L. Brewer, who heated known mixtures of the elements and submitted them to us for X-ray examination. A new phase was found whose composition corresponded to $MoSi_{0{\cdot}35 \pm 0{\cdot}05}$. Powder-diffraction patterns of this phase were obtained using copper $K\alpha$ X-rays with a 9 cm. diameter powder camera and with a 'Norelco' spectrometer. The density of a small sample was determined by measurement of its apparent weights in air and in toluene. From these data it was possible to deduce the complete structure by a straightforward procedure depending only on the reflections of zero intensity. The result was checked by computation of the intensities of the observed lines.

The diffraction patterns corresponded to a primitive cubic lattice with

$$a = 4{\cdot}890 \pm 0{\cdot}002 \text{ A}.$$

The density measured by buoyancy was $8{\cdot}4 \pm 0{\cdot}3$ g.cm.$^{-3}$, where the uncertainty is due to the weighing error of the small sample used. This value corresponds to a molecular weight of 592. The composition $MoSi_{0{\cdot}35 \pm 0{\cdot}05}$ deduced from the phase studies corresponds to 91 ± 1 % molybdenum. Thus there are $5{\cdot}6 \pm 0{\cdot}3$, i.e. 6 molybdenum atoms, and $2{\cdot}1 \pm 0{\cdot}3$, or 2 silicon atoms in each unit cell. For Mo_6Si_2 the density calculated from the X-ray measurement is $8{\cdot}97 \pm 0{\cdot}01$ g.cm.$^{-3}$. The difference from the measured value is not regarded as significant, because of the porous appearance of the sample tested by buoyancy.

Reflections were observed for planes hhl only if $l = 2n$. Also absent were 410, 430 and 531. All other lines up to $h^2 + k^2 + l^2 = 38$ were observed. These extinctions limit the space group to O_h^3–$Pm3n$ and T_d^4–$P\bar{4}3n$, which have sets of 2, 6, 8, 12 and more equivalent positions. The twofold and sixfold special positions for these two space groups are identical, so it is sufficient to consider O_h^3. The two silicon atoms must be in 2 (a): $0,0,0;\ \tfrac{1}{2},\tfrac{1}{2},\tfrac{1}{2}$. Molybdenum in 6 ($b$): $0,\tfrac{1}{2},\tfrac{1}{2};\ \tfrac{1}{2},0,\tfrac{1}{2};\ \tfrac{1}{2},\tfrac{1}{2},0;\ 0,0,\tfrac{1}{2};\ 0,\tfrac{1}{2},0;\ \tfrac{1}{2},0,0$ does not permit the observed reflections 210, 320, 421, and others. The two remaining sets 6 (c) and 6 (d) differ by the translation $\tfrac{1}{2}, \tfrac{1}{2}, \tfrac{1}{2}$, and therefore give equivalent structures when combined with 2 (a). Such a combination moreover requires all the observed extinctions. Therefore, the structure is:

Space group O_h^3–$Pm3n$.
2 Si in (a): $0,0,0;\ \tfrac{1}{2},\tfrac{1}{2},\tfrac{1}{2}$.
6 Mo in (c): $\tfrac{1}{4},0,\tfrac{1}{2};\ \tfrac{1}{2},\tfrac{1}{4},0;\ 0,\tfrac{1}{2},\tfrac{1}{4};\ \tfrac{3}{4},0,\tfrac{1}{2};\ \tfrac{1}{2},\tfrac{3}{4},0;\ 0,\tfrac{1}{2},\tfrac{3}{4}$.

Intensities were calculated for this structure by the equation

$$I = p \mid F_{hkl} \mid^2 \frac{1 + \cos^2 2\theta}{\sin^2\theta \cos\theta} \times 3{\cdot}3 \times 10^{-5},$$

where F_{hkl} is the structure factor, p is the multiplicity, and θ is the Bragg angle. The numerical factor reduces the intensities to the arbitrary scale of the spectrometer values. The agreement of these intensities with the observed values listed in Table 1 confirms the structure deduced above. Reflections required to be absent by the symmetry are omitted from the table. The slightly low values observed for the first few lines are probably the result of absorption in the sample.

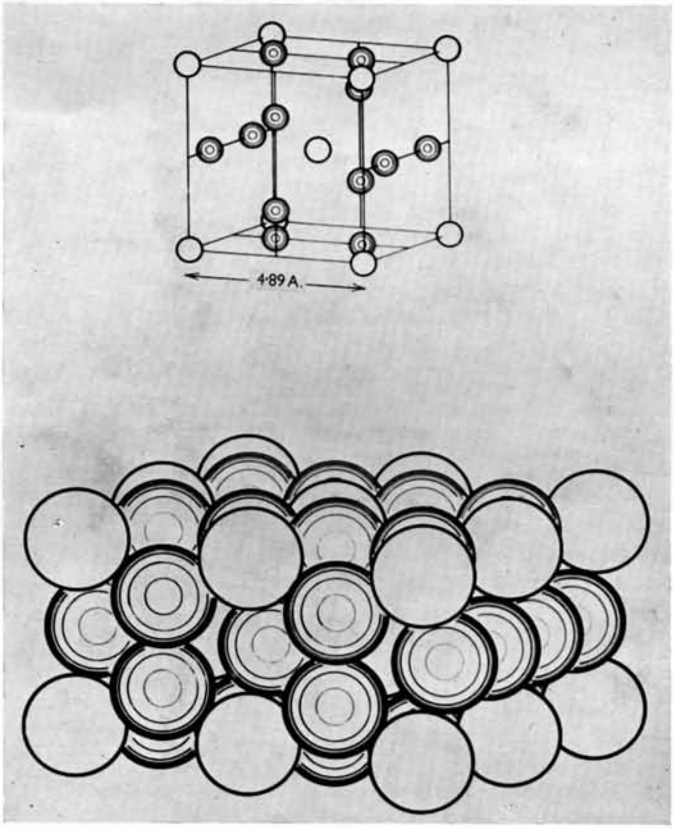

Fig. 1. Structure of Mo_3Si. Above, one unit cell; below, four unit cells, with atoms approximately to scale. The molybdenum atoms are shaded.

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†	 Spectrometer	 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

* $a=4{\cdot}890$; Cu $K\alpha_1=1{\cdot}54050$ 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).

This research was made possible by the close cooperation of A. Searcy and Prof. L. Brewer. We are indebted to Dr K. J. Palmer and Miss M. Ballantyne of Western Regional Research Laboratory, U.S. Department of Agriculture, for the spectrometer record. Mrs L. Jackson obtained the photographic diffraction patterns. This research was supported by the United States Atomic Energy Commission.

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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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