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Two unconventional uses of a Weissenberg goniometer. By E. J. W. WHITTAKER, *Technical Division, Ferodo Ltd, Chapel-en-le-Frith, Stockport, England*

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Although the essential feature of a Weissenberg goniometer is the coupling of crystal rotation with film translation, certain of its other characteristics can be used to considerable advantage in circumstances when an ordinary Weissenberg photograph is not desired or cannot be obtained.

The latter circumstance arises in the investigation of those fibrous materials which give a rotation photograph even when the specimen is stationary. Hitherto the diffraction data obtained from such materials have been limited to the region of reciprocal space which can be explored by means of a single rotation photograph, since it is not possible to obtain useful diffraction photographs by rotation or oscillation about any axis other than the fibre axis. By use of shorter wavelength radiation, it is of course possible to extend the volume of reciprocal space within the cone of reflexion at the cost of reduced resolution of the photograph, but even by this means the important central regions of the higher layers of the reciprocal lattice remain inaccessible in the hollow of the cone. The difficulty can be overcome by applying to the rotation photograph, the equi-inclination method which is well known in connection with Weissenberg photographs. Although all the refinements of a Weissenberg goniometer are not required for this purpose, it is a convenient instrument to use (without a layer-line screen) since an ordinary X-ray goniometer is not adapted for operation with the beam inclined to the camera axis.

The equi-inclination rotation photograph has two merits: it records the whole of the chosen layer line between $\xi = 0$ and the boundary of the limiting sphere, and for any given radiation it approximately doubles the number of layer lines which can be conveniently recorded. If the maximum inclination of the instrument is in the neighbourhood of 40° it is convenient to record layer lines up to $\zeta = 1.4$ whereas the practical limit with the normal-beam arrangement is $\zeta = 0.7$. The only structural alterations to a standard Weissenberg goniometer which are required for this purpose are the removal of the layer-line screen and the provision of a pivoted beam trap. The usual elongated slot is unsatisfactory for this purpose since it obscures too much of the photograph, but it is a simple matter to provide a cylindrical

beam trap which can be adjusted to be coaxial with the collimator after setting the inclination of the camera.

The second technique makes use of the fact that a Weissenberg goniometer has provision for oscillating the film holder over any desired distance. If this oscillatory motion is restricted to an amplitude of a few millimetres, and freed from its usual synchronism with the crystal oscillation or rotation, then each spot on the resulting photograph is drawn out into a short streak parallel to the camera axis and perpendicular to the layer line on which it lies. The central portion of this streak will be of uniform intensity in the direction perpendicular to the layer line, for precisely the same reasons as hold for the central region of a spot produced by an integrating Weissenberg instrument (Wiebenga & Smits, 1950). Accordingly it is possible to obtain true spot profiles in the direction of the layer line, using an ordinary microdensitometer. This technique has proved useful in studying the diffuse scattering along the layer lines in disordered fibrous structures such as chrysolite which could not be satisfactorily investigated by any other means. It could also be applied to single-crystal oscillation photographs, provided that the ratio of the period of oscillation of the crystal to that of the camera was made equal to the ratio of two sufficiently large relatively prime numbers. This semi-integrating technique therefore provides a method of obtaining instrumental intensity data from single crystals using only an ordinary Weissenberg goniometer and a microdensitometer. It is, of course, more time-consuming than the integrating Weissenberg technique, but has the merit of using apparatus which is more generally available.

It is scarcely necessary to add that the technique of the equi-inclination rotation photograph and the semi-integrating rotation photograph may be combined without losing any of the advantages of either.

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Reference

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The crystal structure of the intermetallic compound $Mg_6Si_7Cu_{16}$.* By GUNNAR BERGMAN and JOHN L. T. WAUGH, *Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena 4, California, U.S.A.*

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A ternary compound in the magnesium-silicon-copper system has been studied by Witte (1938) and given the approximate formula $Mg_6Si_7Cu_{16}$ by him. The X-ray investigation he carried out showed that the compound

has a face-centered cubic structure with $a_0 = 11.67 \pm 0.01 \text{ \AA}$ and with the calculated number of atoms per unit cube equal to 116. Witte also found the Laue symmetry to be O_h . He did not determine the position of the atoms.

We have now determined the crystal structure by the stochastic method, and have verified the structure with X-ray data obtained from single crystals. The space

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Table 1. *Description of structure*

No. of Atoms	Position	<i>x</i>	<i>y</i>	<i>z</i>	Kind	Ligancy
4	(b)	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	Si	14
24	(d)	$\frac{1}{2}$	$\frac{1}{2}$	0	Si	12
24	(e)	0.179	0	0	Mg	17
32	(f)	0.169	0.169	0.169	Cu	12
32	(f)	0.376	0.376	0.376	Cu	13

group is $O_h^5-Fm\bar{3}m$. The structure can be described as follows. At (0, 0, 0) there is a group of magnesium atoms with the atoms located at the vertices of an octahedron. Outside this group of atoms there are eight copper atoms at the vertices of a cube. Twelve silicon atoms at the vertices of a cubo-octahedron surround this cluster of fourteen atoms. The twelve silicon atoms are shared with the clusters associated with the neighboring lattice points. At $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ there is finally one silicon atom surrounded by eight copper atoms at the vertices of a cube. There are consequently six magnesium atoms, seven silicon atoms, and sixteen copper atoms per lattice point and 116 atoms per unit cube, in agreement with Witte's findings.

Although the structure factors calculated for this structure agreed well with the observed ones it could not be taken as certain that the copper and silicon atoms are not disordered to some extent, as copper and silicon are known to form binary phases with disordered crystal structures, e.g. Cu_5Si . Admittedly disorder appeared very unlikely since the phase is known to occur with a composition that is always very close to the composition of the ordered structure described above. Fourier projections showed, however, that the structure is most likely completely ordered.

With the notation in the *International Tables for the*

Determination of Crystal Structures (1935) for space groups $O_h^5-Fm\bar{3}m$ the structure may be described as in Table 1.

The parameters are obtained from a least-squares refinement based on $hk0$ data obtained with $CuK\alpha$ radiation.

Florio, Rundle & Snow (1952) have recently reported on the determination of the crystal structure of the intermetallic compound Th_6Mn_{23} . The structure they found is a simpler variation of the structure described above. The thorium atoms occupy the same positions as the magnesium atoms and the manganese atoms occupy the same positions as the silicon and copper atoms in the ternary phase.

A detailed report on the determination of the $Mg_6Si_7Cu_{16}$ structure will be published later.

The authors wish to thank Prof. Linus Pauling for suggesting this problem and for helpful criticism.

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Silver formed by electron bombardment of silver bromide. By CHESTER R. BERRY and ROBERT L. GRIFFITH, *Eastman Kodak Company, Rochester, N. Y., U. S. A.*

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In his recent publication on the growth of silver which occurs during electron bombardment of thin films of oriented silver bromide, Trillat (1952) states that in our experiments (Berry & Griffiths, 1950) on the decomposition of silver bromide by light, the important action of electrons on the liberation of silver does not appear to have been considered sufficiently. On the contrary, we were quite aware of the possible photographic action on the crystals of the detecting beams of both X-rays and electrons, and we reported in our paper some measurements on the effect in both cases. Confining our attention to one of our statements on the action of electron beams, we said: 'Before ultraviolet irradiation, no silver was detected, even after electron bombardment for several times the normal exposure'. The time of the electron bombardment used in these tests was of the order of 100 times that used in the regular diffraction experiments. We would not deny that some silver was produced by the measuring electron beam, but the amount was certainly small compared with that which was produced by the

ultraviolet and visible illumination. Whatever silver was produced by the electrons would doubtless add to the large crystals already produced by the light. We feel confident that the orientations of the large quantities of silver produced by the light were in no way altered by our measuring beams of X-rays or electrons.

We also wish to comment on Trillat's observation that no preferred orientation of silver was produced by the action of photographic developer on his evaporated films. We have found that the parallel orientation of silver may be produced on large single crystals of silver bromide by the action of Kodak Developer D-19, diluted to about 1/1000 normal strength, but that twinning and random orientation are found in the silver produced by more rapid reaction.

References

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