

analysis, this error will be important only if the double reflexion caused by two strongly reflecting lattice planes coincides with a very weak (or absent) reflexion. The occurrence of this effect can be recognized in single-crystal diagrams taken with unfiltered radiation: if—with the given crystal setting—the azimuth conditions for double reflexion via a certain pair of lattice planes are fulfilled for the α radiation, this will not hold for the β radiation. So weak reflexions whose intensities are enhanced by double reflexion can be unmasked by a comparison of their α and β components.

The author wishes to thank Prof. J. M. Bijvoet for his stimulating interest and advice in the course of this investigation.

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On the Structure of Uranium in Thin Film

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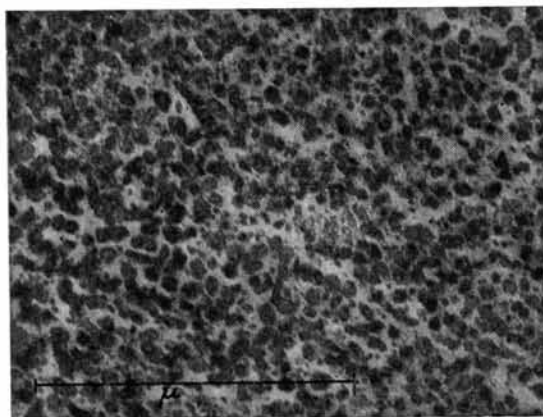
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Thin film of uranium, prepared by the vacuum evaporation technique, was studied with the help of electron microscopy and electron diffraction. The analysis shows that uranium in thin film assumes the β -phase structure (tetragonal; $a = 10.52$, $c = 5.57$ Å; $P4nm$).

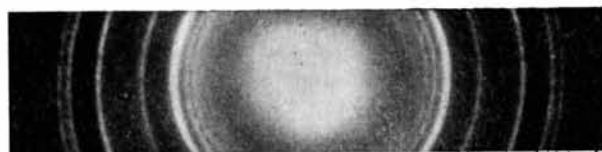
X-ray crystallographic study of uranium has been done at different temperatures by different authors (Jacob & Warren, 1937; Tucker, 1951; Wilson & Rundle, 1949). It is found that uranium assumes the orthorhombic structure (α -phase; $a = 2.852$, $b = 5.865$, $c = 4.945$ Å and space group $Amam$) up to 660 °C., tetragonal (β -phase; $a = 10.52$, $c = 5.57$ Å and space group $P4nm$) between 660 °C. and 760 °C. and body-centred cubic (γ -phase; $a = 3.474 \pm 0.005$ Å and space group $Im3m$) between 760 °C. and the melting point. In the present work investigation is made on the structure of uranium in thin film with the help of electron microscopy and electron diffraction technique.

Thin film of uranium was prepared by the evaporation of a weighed quantity of the metal from a tungsten bucket inside a vacuum chamber (pressure 10^{-4} mm.Hg.). The evaporated metal was allowed to deposit on a collodion film covering a steel wire-mesh. Fig. 1(a) is the electron micrograph ($54,000\times$) of the uranium film taken with an accelerating potential of 60 kV. using the Siemen's Elmiskop I. The micrograph shows the uranium microcrystals well resolved. Fig. 1(b) is the electron diffraction photograph of the film. The diffraction length was 587 mm. and the accelerating potential 60 kV. The indexing of the diffraction rings is shown in the figure. The diffraction pattern is found to correspond with the tetragonal structure (β -phase) of uranium. Table I shows the

agreement between the theoretical and experimental ring diameters and also the relative intensities of the



(a)



(b)

Fig. 1. (a) Electron micrograph of the uranium film. (b) Electron diffraction photograph of the uranium film.

Table 1. *Analysis of the uranium diffraction pattern*

<i>hkl</i>	Estimated diameters in mm.	Measured diameters in mm.	Intensity
311	19.75	19.75	<i>vf</i>
330	22.60	22.50	<i>f</i>
212	24.50	24.25	<i>f</i>
312	26.40	26.50	<i>vs</i>
620	33.00	33.00	<i>s</i>
611	34.20	34.50	<i>vf</i>
114	41.10	41.50	<i>vs</i>
424	47.00	47.00	<i>s</i>
434	48.50	48.30	<i>s</i>
115	51.00	51.00	<i>vf</i>
860	53.60	53.50	<i>vf</i>
084	58.80	58.50	<i>f</i>

v = very; *f* = faint; *s* = strong.

rings. Also in the electron micrograph (Fig. 1(a)) some tetragonal crystals can be identified.

In the thin film state uranium thus assumes the β -phase structure which according to X-ray studies should occur at a high temperature. It may be pointed out that the heating of the specimen during the examination in the electron microscope might be responsible for the phase transformation. In this respect special care was taken to keep the intensity of the electron beam at a minimum. The experiment

was repeated with different films prepared under the same condition but the result was always the same. Similar structural anomalies in the thin film state have been observed by other authors in several cases. Quarrel (1937) reported that many metals which normally crystallize as face-centred cubes were found to develop the hexagonal close-packed structure in the thin film. Aggarwal & Goswami (1957) observed very recently a new phase (face-centred cube) of molybdenum in thin film. The mechanism of such phase transformations in the thin film state is not yet clearly understood and hence deserves special attention.

The author is indebted to Prof. N. N. Das Gupta for his kind interest in the present work.

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A Detailed Refinement of the Crystal Structure of Potassium Ethyl Sulphate

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Refinement of the crystal structure of potassium ethyl sulphate using Jarvis's three-dimensional data has been carried out until $R = 0.098$. Anisotropic thermal parameters have been determined, and an estimate has been made of the error arising from rotational oscillation. The bond lengths are $S-O_1 = 1.466$ Å, $S-O_2 = 1.466$ Å, $S-O_3 = 1.451$ Å (mean $S-O = 1.46 \pm 0.01$ Å), $S-O_4 = 1.60 \pm 0.01$ Å, $C-O = 1.45 \pm 0.02$ Å and $C-C = 1.51 \pm 0.02$ Å.

Introduction

In most compounds in which oxygen is bonded only to sulphur the S-O bond lengths are close to 1.43 Å (see Abrahams, 1956 for a summary); it is probable, therefore, that 1.43 Å is the double-bond length. The S-O bond may be longer, about 1.6 Å, when the oxygen is bonded to another atom as in $K_2S_2O_7$ (Lynton, 1955) and the ion $HS_2O_7^-$ (Steeman & MacGillavry, 1954) but these interatomic distances have not been determined accurately. Jarvis (1953) studied the structure of potassium ethyl sulphate, $KO_3SOC_2H_5$, by three-dimensional X-ray crystal structure analysis and found that the three chemically equivalent S=O

bonds were 1.44, 1.45 and 1.49 Å while the S-O bond to the esterified oxygen was 1.60 Å. Jarvis's results were obtained after one cycle of correction for series

Table 1. *Electron densities and curvatures*
Ratios between observed and calculated values

	ρ	$\partial^2\rho/\partial x^2$	$\partial^2\rho/\partial y^2$	$\partial^2\rho/\partial z^2$
K ⁺	0.976	1.005	0.952	0.911
S	0.998	1.027	0.987	0.943
O ₁	0.975	0.950	1.020	0.990
O ₂	1.023	1.013	1.026	1.011
O ₃	0.972	1.035	0.906	0.883
O ₄	1.000	1.008	1.016	1.018
C ₁	0.900	0.874	0.779	0.868
C ₂	0.871	0.862	0.720	0.890