all atoms in the larger crystal II having smaller thermal parameters than the corresponding atoms in crystal I. This systematic difference is presumably largely caused by the extinction in crystal II being larger than that in crystal I; to a smaller extent systematic errors in the absorption corrections may play a part (Jellinek, 1958).

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The crystal structure of niobium monoxide.*

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The crystal structure of niobium monoxide has been described as cubic, space group *Pm*3*m*, with three niobium atoms in 3(*c*) ($0\frac{1}{2}\frac{1}{2}$; $\frac{1}{2}0\frac{1}{2}$; $\frac{1}{2}\frac{1}{2}0$) and three oxygen atoms in 3(*d*) ($\frac{1}{2}00$; $0\frac{1}{2}0$; $00\frac{1}{2}$), (Brauer, 1941; Andersson & Magnéli, 1957) on the basis of X-ray powder diffraction data. However, only the niobium positions could be determined with certainty because of the relatively small X-ray scattering power of oxygen. We have now confirmed the oxygen positions by neutron diffraction.

Niobium monoxide was prepared by heating a stoichiometric mixture of niobium and Nb_2O_5 powders for 1 hour at 1700° under vacuum of 5×10^{-6} torr or better. A lattice constant of $4{\cdot}2101 \pm 0{\cdot}0003$ Å was determined from X-ray powder diffraction data (copper $K\alpha$, $\lambda = 1{\cdot}54051$ Å). The neutron-diffraction data were obtained at the Los Alamos Omega West reactor with a monochromatic beam, $\lambda = 1{\cdot}1357$ Å and with a step increment of 0·2° in 2θ. The sample was contained in a parallel-sided holder made from a null-matrix alloy (Ti–Zr) (Sidhu, Heaton, Zauberis & Campos, 1956), and was placed in the symmetrical transmission position. The neutron and X-ray diffraction traces are shown in Fig. 1.

The observed neutron intensities were determined by least-squares analysis of the diffraction data (Bowman, Wallace, Yarnell, Wenzel & Storms, 1965), and were fitted to the trial structure by least-squares solution of the equation (Bacon, 1962)

$$I = K \frac{\exp(-\mu t \sec \theta)}{\sin^2 2\theta} \exp\left(-2B \frac{\sin^2 \theta}{\lambda^2}\right) jF^2 ,$$

with $K = 0{\cdot}265 \pm 0{\cdot}002$, $B = 0{\cdot}34 \pm 0{\cdot}02$, $R = 0{\cdot}025$, where $R = \Sigma\, w|I_o - I_c| / \Sigma\, wI_o$. Neutron scattering lengths of $0{\cdot}691 \times 10^{-12}$ cm for niobium and $0{\cdot}577 \times 10^{-12}$ cm for oxygen were used. The observed and calculated neutron diffraction intensities are compared in Table 1. The excellent agreement fully confirms the previously assigned oxygen positions.

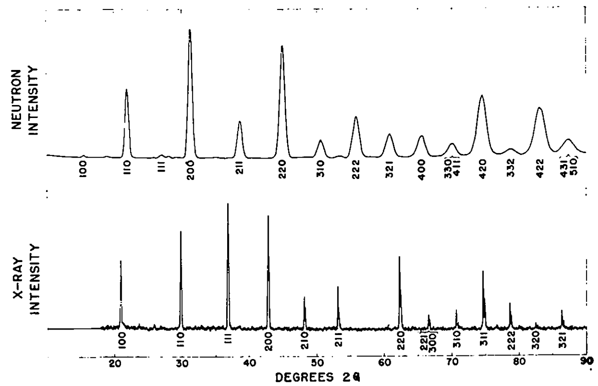

Fig. 1. Neutron ($\lambda = 1{\cdot}1357$ Å) and X-ray ($\lambda = 1{\cdot}54051$ Å) diffraction patterns of NbO.

Table 1. *Summary of experimental data*

hkl	*d*	*I* (obs)	*I* (calc)
100	4·210	< 0·3	0·3
110	2·977	31·6	31·2
111	2·431	1·4	1·0
200	2·105	71·0	71·1
210	1·833	< 0·3	0·2
211	1·719	22·4	21·4
220	1·489	73·9	73·7
221 300	1·403	< 0·3	0·2
310	1·331	12·5	13·4
311	1·269	< 0·3	0·9
222	1·215	33·2	34·2
320	1·168	< 0·3	0·1
321	1·125	20·6	20·0
400	1·053	20·4	20·3
410	1·021	< 0·3	0·1
411 330	0·992	11·8	12·4
331	0·966	< 0·3	0·6
420	0·941	68·1	68·9
421	0·919	< 0·3	0·1
332	0·898	5·5	7·2
422	0·859	62·7	61·8
500 430	0·842	< 0·3	0·0
510 431	0·826	20·3	19·8

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