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The Structure of Nb_6Sn_5*

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The crystal structure of the intermetallic compound Nb_6Sn_5 has been determined. The space-group symmetry was found to be *Immm* (D_{2h}^{25}), and lattice parameters were measured as $a = 5{\cdot}656 \pm 0{\cdot}002$, $b = 9{\cdot}199 \pm 0{\cdot}003$, $c = 16{\cdot}843 \pm 0{\cdot}004$ Å. The compound shows pronounced layering normal to the short axis.

Introduction

Interest in niobium–tin alloys stems from the discovery of the structure and superconducting behavior of Nb_3Sn (Matthias, Geballe, Geller & Corenzwit, 1954; Geller, Matthias & Goldstein, 1955). A number of studies of the phase relationships in the system have been made (Agafonova, Baron & Savitskii, 1959; Wyman, Cuthill, Moore, Park & Yakowitz, 1962; Reed, Gatos, Lafleur & Roddy, 1962; Ellis & Wilhelm, 1964; Enstrom, Pearsall & Wulff, 1964; Schadler & Rosenbaum, 1964). Even though there is significant disagreement among the various proposed phase diagrams, nonetheless there is, with the single exception of the work of Agafonova, Baron & Savitskii, general agreement that two compounds exist on the tin-rich side of Nb_3Sn. Both of these compounds have been reported to be superconducting; Reed & Gatos (1962) found a critical temperature of 16·6°K for 'Nb_3Sn_2' while van

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Ooijen, van Vucht & Druyvesten (1962) found a critical temperature of 2·60°K for $NbSn_2$. Single crystals of these two compounds suitable for X-ray diffraction studies were obtained from alloys used in the investigation of Ellis & Wilhelm.

The compound which is most rich in tin was found to crystallize in the orthorhombic space group *Fddd* (D_{2h}^{24}) with lattice constants $a=5{\cdot}655\pm0{\cdot}001$; $b=9{\cdot}860\pm0{\cdot}001$, $c=19{\cdot}152\pm0{\cdot}004$ Å. Intensity data from a single crystal of this phase indicate that the crystal structure is isomorphous with the Mg_2Cu structure reported by Schubert & Anderko (1951). The ideal stoichiometry of the phase is thus indicated to be $NbSn_2$, though Schadler & Rosenbaum (1964) report that this phase and the other phases in the system have a range of homogeneity. Since the $NbSn_2$ structure has previously been published (Gomes de Mesquita, Langereis & Leenhouts, 1963), the present work with respect to this phase represents only a corroboration and further discussion seems unnecessary. The remainder of the paper will therefore be concerned with the second compound whose stoichiometry on the basis of the structure determination is indicated to be Nb_6Sn_5.

Symmetry and cell dimensions

Diffraction patterns, *h0l* through *h5l*, were taken with a Weissenberg camera and Cu *Kα* radiation, and the patterns, *0kl* through *3kl* and *hk0* through *hk8*, were taken with a precession camera and Mo *Kα* radiation. In every case C_{2l} symmetry was observed. This indicates an orthorhombic crystal system, and precision lattice parameters were determined from back-reflection Weissenberg patterns taken with Cu *Kα* radiation. Values of $a=5{\cdot}656\pm0{\cdot}002$, $b=9{\cdot}199\pm0{\cdot}003$, and $c=16{\cdot}843\pm0{\cdot}004$ Å were obtained by a least-squares treatment of the data with a weighting factor based on the function proposed by Nelson & Riley (1954). These parameters are in good accord with those reported by Enstrom, Pearsall & Wulff (1964) and those by van Vucht, Bruning & Donkersloot (1963).

Examination of the diffraction patterns showed that reflections occurred with $h+k+l=2n$ and with no other systematic extinctions. The probable space group is thus one of the following four: *Imm*2 (C_{2v}^{20}), *I*222 (D_2^8), $I2_12_12_1$ (D_2^9), or *Immm* (D_{2h}^{25}). Intensity data for 370 independent reflections of the type *hkl* were obtained by visual estimation using a multiple film technique with a Weissenberg camera and Cu *Kα* radiation. These intensity data were analyzed by the method proposed by Howells, Phillips & Rogers (1950), and the intensity distributions are shown in Fig. 1. The results definitely favor the centric space group *Immm* (D_{2h}^{25}).

A three-dimensional Patterson projection was synthesized from the copper intensity data, and trial structures were postulated in the centric space group. However, attempts to refine the structures with the copper data emphasized the severity of the absorption problem which arises from a combination of crystal shape and large mass absorption coefficients. The shape in this case was roughly that of a truncated rectangular prism with edges approximately 0·52 × 0·25 × 0·14 mm, and the crystal axes were determined to be nearly parallel to the prism edges. The mass absorption coefficients for Nb and Sn with Cu *Kα* radiation are respectively 153 $cm^2.g^{-1}$ and 256 $cm^2.g^{-1}$, while with Mo *Kα* radiation they are 17·1 and 31·1 $cm^2.g^{-1}$.

Therefore, for refinement purposes a second set of intensity data was taken with Mo *Kα* radiation using a General Electric Spectrogoniometer with a scintillation counter detector. A total of 596 independent re-

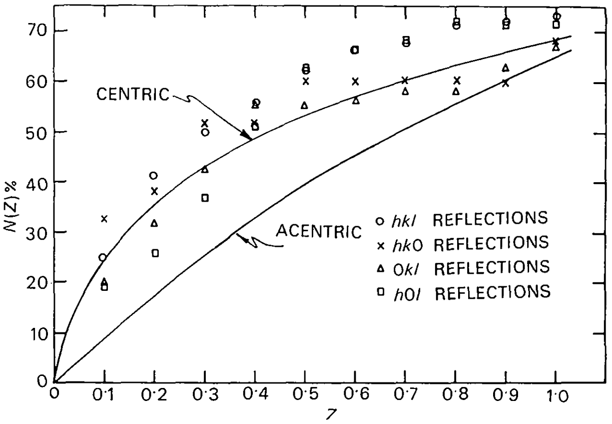

Fig. 1. Intensity distribution with the solid lines giving the theoretical distributions and with $N(Z)$ being the relative number of reflections with an intensity less than or equal to Z % of the local average intensity.

Table 1. *Refined structural parameters for* Nb_6Sn_5

Atom type	Wykoff notation	x_n	y_n	z_n	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	B_n(Å^2)
Nb	8(*k*)	0·2500	0·2500	0·2500	—	—	—	0·18 ± 0·03
Nb	8(*l*)	0·0000	0·2730	0·0853	—	0·0004	0·0003	0·29 ± 0·04
Nb	4(*i*)	0·0000	0·0000	0·3469	—	—	0·0004	0·33 ± 0·07
Nb	4(*e*)	0·236	0·0000	0·0000	0·002	—	—	0·25 ± 0·06
Sn	8(*l*)	0·0000	0·3073	0·3957	—	0·0003	0·0002	0·29 ± 0·03
Sn	4(*f*)	0·256	0·5000	0·0000	0·002	—	—	0·22 ± 0·04
Sn	4(*j*)	0·5000	0·0000	0·7061	—	—	0·0003	0·22 ± 0·04
3·68 Sn or (2·22 Sn and 1·78 Nb)	4(*i*)	0·0000	0·0000	0·1682	—	—	0·0003	0·12 ± 0·04

Table 2. *Comparison of observed and calculated structure factors*

hkℓ	$\lvert F_o \rvert$	F_c
0,0,2	3.1	-2.1
0,0,4	5.3	3.1
0,0,6	17.4	-15.5
0,0,8	36.8	36.3
0,0,10	37.1	35.3
0,0,12	50.3	56.6
0,0,14	7.1	-5.4
0,0,16	3.6	-0.3
0,0,18	9.0	8.1
0,0,20	39.6	40.7
0,0,22	7.7	-7.3
0,0,24	27.5	27.1
0,0,26	5.6	-3.7
0,0,28	18.0	19.5
0,0,30	5.7	6.1
0,0,32	6.9	4.0
0,0,34	<3.9	1.9
0,0,36	13.9	14.7
0,0,38	8.5	7.4
0,0,40	10.2	10.6
0,0,42	5.4	-6.5
0,0,44	3.1	4.1
0,1,1	4.8	-2.0
0,1,3	<9.2	1.4
0,1,5	24.6	-19.7
0,1,7	9.1	7.3
0,1,9	9.9	-7.9
0,1,11	12.7	10.9
0,1,13	8.7	-8.4
0,1,15	23.4	-23.7
0,1,17	19.1	19.5
0,1,19	4.0	-4.7
0,1,21	5.4	-5.3
0,1,23	<3.9	2.4
0,1,25	9.7	-9.9
0,1,27	<4.0	-2.7
0,1,29	7.0	4.5
0,1,31	3.0	4.3
0,1,33	12.5	-13.0
0,1,35	<3.8	0.1
0,1,37	3.9	4.3
0,1,39	6.5	-7.8
0,1,41	5.0	5.1
0,1,43	2.4	-3.0
0,1,45	5.3	-5.3
0,2,0	9.0	-1.3
0,2,2	12.3	10.4
0,2,4	31.1	27.0
0,2,6	68.0	106.6
0,2,8	14.9	-12.0
0,2,10	10.2	9.2
0,2,12	9.6	-9.0
0,2,14	44.3	48.2
0,2,16	6.0	5.2
0,2,18	36.5	35.7
0,2,20	<3.8	0.7
0,2,22	6.7	6.2
0,2,24	12.5	13.3
0,2,26	17.5	15.2
0,2,28	<4.0	-1.6
0,2,30	19.4	20.8
0,2,32	<4.0	0.3
0,2,34	16.5	18.8
0,2,36	3.6	-5.2
0,2,38	5.4	5.6
0,2,40	<3.1	2.8
0,2,42	7.5	15.1
0,2,44	4.0	3.8
0,3,1	23.2	-20.1
0,3,3	17.9	14.4
0,3,5	8.7	5.3
0,3,7	<3.8	1.7
0,3,9	36.5	-33.8
0,3,11	<3.7	2.5
0,3,13	14.4	13.8
0,3,15	<3.7	-3.3
0,3,17	9.3	9.4
0,3,19	26.9	-28.1
0,3,21	11.7	-10.2
0,3,23	20.3	20.1
0,3,25	5.7	4.9
0,3,27	10.7	-9.3
0,3,29	10.0	-11.5
0,3,31	<4.0	0.8
0,3,33	<4.0	-2.1
0,3,35	9.3	8.4
0,3,37	<3.6	0.3
0,3,39	14.9	-15.9
0,3,41	2.7	4.1
0,3,43	<2.4	2.5
0,4,0	61.8	105.7
0,4,2	12.9	-9.5
0,4,4	25.7	24.3
0,4,6	10.8	5.8
0,4,8	26.3	22.3
0,4,10	16.3	11.6
0,4,12	48.5	48.8
0,4,14	10.1	9.8
0,4,16	8.6	8.5
0,4,18	<3.9	-0.1
0,4,20	28.9	28.0
0,4,22	5.7	-4.9
0,4,24	34.3	34.9
0,4,26	<4.1	-1.5
0,4,28	10.8	11.9
0,4,30	<4.1	1.3
0,4,32	9.8	7.7
0,4,34	6.7	7.6
0,4,36	13.3	13.5
0,4,38	<3.4	1.6
0,4,40	7.3	8.4
0,4,42	<2.6	-2.4
0,4,44	5.9	7.6
0,5,1	<4.7	2.4
0,5,3	9.2	-5.3
0,5,5	24.7	-22.0
0,5,7	14.0	11.4
0,5,9	5.7	4.3
0,5,11	11.9	9.7
0,5,13	21.1	-19.8
0,5,15	31.0	-29.9
0,5,17	23.9	24.7
0,5,19	8.9	8.5
0,5,21	<4.0	-3.1
0,5,23	8.1	-8.3
0,5,25	18.8	-17.5
0,5,27	<4.1	1.2
0,5,29	14.8	13.3
0,5,31	7.0	6.5
0,5,33	16.3	-18.1
0,5,35	5.6	-5.1
0,5,37	4.3	5.3
0,5,39	<3.1	-3.1
0,5,41	6.0	6.3
0,5,43	4.3	-5.2
0,6,0	36.1	36.0
0,6,2	20.2	17.4
0,6,4	11.7	-8.5
0,6,6	52.0	55.9
0,6,8	7.5	1.9
0,6,10	37.5	34.9
0,6,12	<4.1	-3.3
0,6,14	23.1	21.2
0,6,16	9.0	-8.0
0,6,18	43.2	41.4
0,6,20	15.2	14.8
0,6,22	<4.1	3.2
0,6,24	3.3	-0.2
0,6,26	10.2	10.9
0,6,28	8.3	7.9
0,6,30	24.9	25.6
0,6,32	4.1	-5.5
0,6,34	7.7	9.5
0,6,36	<3.6	3.3
0,6,38	12.6	13.2
0,6,40	4.0	5.2
0,6,42	5.4	8.4
0,7,1	<5.2	-3.5
0,7,3	8.8	6.8
0,7,5	10.9	-8.0
0,7,7	6.5	-4.3
0,7,9	24.8	-21.3
0,7,11	11.2	10.1
0,7,13	14.4	12.4
0,7,15	8.2	-8.2
0,7,17	5.0	4.3
0,7,19	23.4	-23.3
0,7,21	5.9	-6.8
0,7,23	18.7	18.2
0,7,25	<4.2	4.0
0,7,27	7.2	-8.2
0,7,29	10.9	-10.3
0,7,31	<4.0	-0.5
0,7,33	<3.9	-3.3
0,7,35	9.7	8.4
0,7,37	<3.3	2.5
0,7,39	11.8	-13.9
0,7,41	<2.5	2.1
0,7,43	<2.0	0.2
0,8,0	45.4	45.5
0,8,2	19.4	-15.4
0,8,4	35.8	39.0
0,8,6	30.5	25.1
0,8,8	11.5	9.0
0,8,10	16.1	-12.1
0,8,12	37.9	33.9
0,8,14	23.0	22.9
0,8,16	19.4	17.6
0,8,18	6.6	-6.0
0,8,20	14.7	13.2
0,8,22	<4.2	-3.4
0,8,24	38.2	39.1
0,8,26	<4.2	0.5
0,8,28	<4.2	4.0
0,8,30	<4.1	-2.6
0,8,32	11.8	11.5
0,8,34	10.5	12.6
0,8,36	11.3	10.5
0,8,38	4.2	-5.0
0,8,40	4.5	6.1
0,8,42	3.4	3.0
0,9,1	20.5	-16.8
0,9,3	<5.6	2.9
0,9,5	6.5	5.5
0,9,7	16.7	13.8
0,9,9	15.7	-13.7
0,9,11	5.7	-5.5
0,9,13	9.9	-8.3
0,9,15	10.7	-10.5
0,9,17	20.8	20.6
0,9,19	<4.3	-4.0
0,9,21	7.5	-7.9
0,9,23	<4.3	-0.2
0,9,25	10.5	-8.2
0,9,27	<4.2	-2.3
0,9,29	7.1	4.3
0,9,31	5.6	5.7
0,9,33	9.2	-9.7
0,9,35	<3.5	-1.7
0,9,37	<3.1	0.1
0,9,39	6.1	-7.8
0,9,41	6.3	7.5
0,10,0	37.2	37.8
0,10,2	19.4	16.8
0,10,4	18.9	-14.2
0,10,6	29.7	30.7
0,10,8	<5.1	1.4
0,10,10	39.4	34.7
0,10,12	6.6	2.9
0,10,14	15.1	13.7
0,10,16	13.5	-11.5
0,10,18	35.6	32.3
0,10,20	16.0	14.1
0,10,22	<4.4	4.4
0,10,24	<4.3	0.2
0,10,26	8.5	9.0
0,10,28	7.2	7.0
0,10,30	21.2	21.6
0,10,32	3.9	-6.5
0,10,34	5.5	8.1
0,10,36	<3.2	-0.9
0,10,38	11.6	13.6
0,10,40	4.0	3.9
0,11,1	18.5	14.4
0,11,3	<6.0	-3.4
0,11,5	31.5	-26.3
0,11,7	7.7	-6.4
0,11,9	<5.1	0.6
0,11,11	22.5	19.2
0,11,13	<4.8	0.2
0,11,15	24.6	-21.7
0,11,17	<4.6	4.5
0,11,19	6.4	-5.5
0,11,33	11.2	-10.7
0,11,35	<3.2	3.2
0,11,37	6.2	6.9
0,11,39	4.7	-6.4
0,12,0	37.5	34.8
0,12,2	16.7	-11.7
0,12,4	28.8	24.1
0,12,6	22.5	18.3
0,12,8	14.4	12.4
0,12,10	7.3	-4.7
0,12,12	24.4	20.5
0,12,14	9.7	8.7
0,12,16	16.4	13.5
0,12,18	<4.7	3.9
0,12,20	17.1	16.4
0,12,24	25.2	24.1
0,12,28	10.0	9.4
0,12,30	<3.8	3.3
0,12,32	6.2	8.0
0,12,34	4.5	5.6
0,12,36	8.4	7.4
0,13,1	25.5	-20.7
0,13,3	9.1	5.9
0,13,5	18.5	14.6
0,13,7	16.5	11.3
0,13,9	18.6	-20.7
0,13,11	13.9	-10.2
0,13,13	<5.0	0.2
0,13,15	<4.9	0.1
0,13,17	15.9	14.4
0,13,19	10.5	-11.8
0,13,33	<3.2	-3.3
0,14,0	19.5	17.6
0,14,2	9.1	10.2
0,14,4	<6.5	-0.2
0,14,6	33.9	27.5
0,14,8	11.6	-7.8
0,14,10	19.1	15.6
0,14,12	7.5	6.4
0,14,14	25.4	23.3
0,14,16	7.0	-5.5
0,14,18	15.9	13.9
0,14,20	<4.7	0.9
0,14,22	9.0	8.3
0,14,26	10.0	9.7
0,14,28	<3.8	-3.4
0,14,30	10.1	10.6
0,14,32	<3.2	-2.3
0,14,34	11.5	14.7
0,15,1	13.9	9.3
0,15,3	<6.8	-3.1
0,15,5	23.5	-18.2
0,15,7	<6.1	-3.3
0,15,9	<5.8	-0.0
0,15,11	16.5	13.1
0,15,13	<5.3	-1.0
0,15,15	19.7	-17.1
0,15,17	4.9	4.6
0,15,19	<4.7	-3.1
0,15,21	<4.5	-0.9
0,15,25	5.6	-5.1
0,15,27	<3.8	-2.2
0,15,33	8.6	-9.4
0,15,35	<2.3	1.9
0,16,0	35.9	34.1
0,16,2	<7.0	-7.0
0,16,4	11.8	8.6

Table 2 (*cont.*)

hkℓ	|Fo|	Fc	hkℓ	|Fo|	Fc	hkℓ	|Fo|	Fc	hkℓ	|Fo|	Fc
0,16,6	11.2	8.3	0,22,0	14.4	13.9	3,13,0	5.8	6.1	7,11,0	<4.5	-0.5
0,16,8	18.1	16.1	0,22,2	4.1	6.6	3,15,0	<4.6	-0.2	7,13,0	<4.8	3.1
0,16,10	5.4	5.9	0,22,4	<5.4	-3.2	3,17,0	<4.9	-0.7	7,15,0	<5.0	-1.7
0,16,12	14.4	12.2	0,22,6	11.6	11.1	3,19,0	8.8	7.2	7,17,0	<4.9	-1.7
0,16,14	5.2	-5.0	0,22,8	<4.8	-4.2	3,21,0	6.9	-5.2	7,19,0	<4.5	4.6
0,16,16	8.6	7.0	0,22,10	9.6	11.9	3,23,0	7.3	5.3	7,21,0	7.2	5.2
0,16,18	11.7	12.8	0,22,12	3.7	5.5	4,0,0	61.2	105.4	8,0,0	47.7	58.9
0,16,20	23.2	21.3	0,22,14	11.5	11.2	4,0,2	2.5	-2.1	8,0,4	<4.1	0.1
0,16,24	8.2	8.7	0,22,16	6.1	-5.2	4,0,4	2.7	-1.4	8,0,8	15.0	14.4
0,16,28	14.7	15.5	0,22,18	6.2	8.0	4,0,6	10.4	-10.7	8,0,12	25.0	26.8
0,16,30	8.2	9.5	0,22,20	<2.8	1.4	4,0,8	24.5	25.8	8,0,16	<4.5	-0.9
0,16,32	2.2	3.2	0,23,1	4.9	-5.9	4,0,12	39.8	43.9	8,0,24	14.3	16.8
0,17,1	<7.1	-6.8	0,23,3	<5.1	1.3	4,0,16	<4.0	-0.5	8,0,32	<3.3	1.9
0,17,3	<7.0	0.7	0,23,5	<4.8	3.0	4,0,20	32.6	34.5	8,2,0	<4.0	-0.4
0,17,5	<6.7	1.0	0,23,7	<4.5	2.8	4,0,24	22.6	23.6	8,4,0	40.2	44.0
0,17,7	8.9	5.6	0,23,9	12.4	-9.6	4,0,32	<3.9	3.3	8,6,0	16.0	17.3
0,17,9	11.8	-8.8	0,23,11	<3.8	-1.7	4,2,0	<2.5	0.5	8,8,0	24.5	23.6
0,17,11	<5.6	-1.4	0,23,13	<3.5	2.2	4,4,0	54.8	75.2	8,10,0	20.9	21.5
0,17,13	<5.3	-2.4	0,23,15	<3.1	-1.9	4,6,0	27.4	28.0	8,12,0	21.5	21.1
0,17,15	13.3	-6.8	0,24,0	9.8	9.4	4,8,0	37.9	37.2	8,14,0	11.0	10.9
0,17,17	13.6	10.1	0,24,2	4.9	-6.3	4,10,0	31.4	31.9	8,16,0	22.6	23.0
0,17,19	<4.6	-4.3	0,24,4	10.5	11.8	4,12,0	33.8	30.1	8,18,0	7.5	6.3
0,18,0	11.3	9.7	0,24,6	11.2	10.8	4,14,0	16.2	15.4	8,20,0	13.8	15.6
0,18,2	<7.1	6.8	0,24,8	2.6	4.6	4,16,0	32.9	30.6	9,0,3	<4.4	-1.6
0,18,4	<11.8	3.5	0,24,10	2.8	-4.4	4,18,0	15.9	8.6	9,0,9	8.9	-7.7
0,18,6	25.8	21.6	1,0,1	2.5	1.2	4,20,0	20.9	19.9	9,0,27	8.6	-12.2
0,18,8	14.8	-9.5	1,0,3	6.4	-2.8	4,22,0	15.4	12.7	9,1,0	4.4	3.4
0,18,10	7.4	7.8	1,0,5	26.9	36.6				9,3,0	19.6	21.1
0,18,12	5.4	6.9	1,1,0	1.8	-1.9	5,0,5	20.2	20.8	9,5,0	6.5	-7.0
0,18,14	24.8	22.6	1,3,0	35.8	47.3	5,0,15	12.7	12.8	9,7,0	16.9	18.8
0,18,16	<4.8	-3.0	1,5,0	27.5	-27.0	5,0,25	15.1	16.0	9,9,0	<4.8	1.8
0,18,18	7.8	5.8	1,7,0	34.5	33.8	5,1,0	4.9	2.5	9,11,0	6.0	5.8
0,18,20	<4.2	-3.6	1,9,0	5.2	3.9	5,3,0	29.9	32.9	9,13,0	8.4	8.3
0,18,22	7.9	8.7	1,11,0	8.4	5.6	5,5,0	15.3	-14.9	9,15,0	<4.6	3.6
0,18,24	16.2	15.4	1,13,0	9.6	9.0	5,7,0	25.0	27.7	9,17,0	<4.0	3.1
0,18,28	7.0	-6.7	1,15,0	<4.5	2.0	5,9,0	<3.7	-0.1	9,19,0	6.4	7.9
0,18,30	5.4	5.2	1,17,0	<4.9	1.1	5,11,0	7.0	6.7	10,0,0	9.5	15.7
0,19,1	7.0	-3.4	1,19,0	10.3	9.1	5,13,0	6.3	9.7	10,0,10	9.3	12.2
0,19,3	<6.9	1.1	1,21,0	<5.0	-4.1	5,15,0	4.8	3.4	10,0,20	<4.4	-3.7
0,19,5	<6.5	-1.2	1,23,0	3.9	6.6	5,17,0	<5.0	2.6	10,0,30	3.1	3.7
0,19,7	<6.1	1.0	2,0,0	35.1	43.8	5,19,0	8.5	9.3	10,6,0	10.9	13.7
0,19,9	12.7	-9.7	2,0,2	10.5	-8.2	5,21,0	<4.6	-2.4	10,8,0	6.9	-6.4
0,19,11	<5.3	1.8	2,0,4	46.3	-90.5	5,23,0	6.3	6.9	10,10,0	15.2	17.3
0,19,13	<5.0	3.2	2,0,6	19.8	-19.4	6,0,0	23.7	25.6	10,12,0	<4.6	-3.8
0,19,15	8.2	-4.7	2,0,8	38.9	-48.0	6,0,2	5.6	-4.4	10,14,0	7.4	9.0
0,19,17	<4.4	4.2	2,0,10	25.8	27.4	6,0,6	10.9	-10.5	10,16,0	<3.7	2.2
0,19,19	11.7	-9.0	2,0,12	13.7	15.3	6,0,12	9.6	-9.9	10,18,0	2.9	5.3
0,20,0	22.3	21.7	2,0,16	49.9	57.3	6,0,18	<4.4	4.1	11,0,11	<4.6	2.8
0,20,2	<6.7	-6.3	2,2,0	6.9	-6.9	6,0,24	11.1	-10.2	11,11,0	4.3	-1.5
0,20,4	8.0	8.6	2,4,0	14.7	12.0	6,0,30	<4.0	3.7	11,13,0	<3.9	1.3
0,20,6	8.0	8.7	2,6,0	28.9	28.4	6,0,36	7.0	-7.4	11,15,0	<3.4	-2.1
			2,8,0	18.2	-17.5	6,2,0	4.6	-2.6	12,0,0	26.8	33.5
0,20,10	<5.3	2.0	2,10,0	33.9	32.4	6,4,0	7.5	8.3	12,0,4	<4.6	-0.5
0,20,12	8.5	8.0	2,12,0	9.9	-10.1	6,6,0	19.5	20.2	12,0,6	<4.6	-4.1
0,20,14	<4.6	-2.7	2,14,0	17.6	14.1	6,8,0	12.7	11.6	12,0,12	12.3	15.4
0,20,16	4.2	6.8	2,16,0	8.2	1.6	6,10,0	25.4	24.9	12,0,18	<3.9	2.7
0,20,18	9.7	9.7	2,18,0	11.3	7.2	6,12,0	7.7	-7.1	12,0,24	7.7	12.9
0,20,20	15.9	14.9	2,20,0	<5.1	-1.7	6,14,0	13.5	11.9	12,4,0	19.9	25.4
0,20,24	8.3	7.5	2,22,0	14.1	12.0	6,16,0	<5.0	1.9	12,6,0	8.9	10.1
0,21,1	<6.4	3.0	2,24,0	3.3	-7.4	6,18,0	8.5	6.5	12,8,0	12.0	14.1
0,21,3	<6.2	-2.9	3,0,1	2.1	-0.7	6,20,0	<4.6	-1.0	12,10,0	13.2	13.2
0,21,5	5.9	-8.6	3,0,3	5.0	-3.9	6,22,0	10.5	10.6	12,12,0	11.2	13.5
0,21,7	<5.5	1.4	3,0,9	17.0	-16.7	7,0,7	14.2	-15.4	13,0,13	3.6	5.0
0,21,9	<5.1	-0.2	3,0,15	15.0	13.8	7,0,21	9.3	-10.8	14,0,0	6.2	9.8
0,21,11	6.7	5.1	3,1,0	6.9	-6.9	7,0,35	10.6	11.5	14,0,14	<2.9	-1.8
0,21,13	<4.4	-4.1	3,3,0	31.7	33.3	7,1,0	7.1	-6.8	14,6,0	4.5	9.3
0,21,15	13.6	-11.8	3,5,0	26.8	-27.5	7,3,0	16.4	16.7	15,0,15	5.2	6.1
0,21,17	6.5	6.5	3,7,0	28.0	26.1	7,5,0	19.1	-19.5			
0,21,19	<3.4	1.3	3,9,0	7.9	-6.9	7,7,0	15.0	14.6			
0,21,21	<3.1	-1.5	3,11,0	5.2	2.3	7,9,0	7.4	6.6			

flections of the type *hk*0, 0*kl*, and *h*0*l* were measured. Standard corrections were made for Lorentz and polarization factors. Dispersion corrections (Templeton, 1962) were made to the atomic scattering factors to the extent that the real part of the correction was incorporated; the imaginary part of the dispersion correction was neglected because the imaginary contribution was sufficiently small. Absorption corrections were made on an IBM 7074 computer with a program based on the approach of Busing & Levy (1957). Refinement of the trial structure was then undertaken by a least-squares treatment with a program of Busing, Martin & Levy (1962).

Initial refinement on positional parameters, isotropic temperature factors, and scale factor produced a residual, $\Sigma||F_o|-|F_c||/\Sigma|F_o|$, of 19·1% with all data. Comparison of the calculated and observed structure factors showed major discrepancies for the most intense reflections in the front reflection region, the calculated structure factor being in these cases always greater in magnitude than the observed value. These discrepancies can thus be reasonably attributed to extinction effects, and abstracting only the five most intense reflections 026, 040, 204, 400, and 440 reduced the residual to 18·1%. Further refinement was made to adjust the atomic scattering factors since the report of Schadler & Rosenbaum (1964) indicated that the compound existed over a range of homogeneity. While the atom factor refinement was not exhaustive because of the limitations of the available refinement program, the results did indicate that one of the fourfold tin sets was either incompletely occupied or was partially substituted with niobium. The indicated composition is 54·9 at.% niobium for the defect model or 58·5 at.% niobium for the substitutional model. In comparison the ideal stoichiometry corresponds to 54·6 at.% niobium.

A subsequent refinement on positional parameters and temperature factors with the adjusted atomic scattering factors produced a residual of 16·8% with all data or 12·5% with 390 observed reflections. The discrepancy in the residuals between inclusion and exclusion of the unobserved reflections prompted a new determination of the intensities of the 55 reflections with calculated values in excess of the estimated limit of observation. For all 55 such reflections careful examination showed that the intensities were observable. After these 55 intensity measurements were made the residual was 12·9% for a total of 445 observed reflections, and the calculated intensities of the additional 145 unobserved reflections were equal to or less than the estimated observable limit. Thus the 12·9% residual is considered to be valid for the structure, and the structure parameters in the centric space group *Immm* (D_{2h}^{25}) are shown in Table 1. In the table, B_n is an isotropic temperature factor for the atom set and σ is the standard deviationof the indicated coordinate. A comparison of observed and calculated structure factors is shown in Table 2, and the coordination and the interatomic distances are shown in Table 3.

Table 3. *Atomic coordination and bond distances in* Nb_6Sn_5

Atom	Neighbor	Number of neighbors	Distance (Å)
Nb: 8(*k*)	Nb 8(*k*)	2	2·828
	Nb 8(*l*)	2	3·120
	Nb 4(*i*)	2	3·154
	Sn 8(*l*)	2	2·882
	Sn 4(*i*)	2	3·031
	Sn 4(*j*)	2	2·798
Nb: 8(*l*)	Nb 8(*k*)	2	3·120
	Nb 8(*l*)	1	2·875
	Nb 4(*e*)	2	3·189
	Nb4 (*i*)	2	3·696
	Sn 8(*l*)	2	2·940
	Sn 4(*f*)	2	2·920
	Sn 4(*i*)	1	2·874
	Sn 4(*j*)	1	2·916
Nb: 4(*i*)	Nb 8(*k*)	4	3·154
	Nb 8(*l*)	4	3·696
	Sn 8(*l*)	2	2·944
	Sn 4(*f*)	2	2·926
	Sn 4(*i*)	1	3·009
	Sn 4(*j*)	2	2·966
Nb: 4(*e*)	Nb 8(*l*)	4	3·189
	Nb 4(*e*)	1	2·677
		1	2·978
	Sn 8(*l*)	4	2·906
	Sn 4(*i*)	2	3·133
Sn: 8(*l*)	Nb 8(*k*)	2	2·882
	Nb 8(*l*)	2	2·940
	Nb 4(*i*)	2	2·944
	Nb 4(*e*)	2	2·906
	Sn 8(*l*)	1	3·513
		1	3·547
	Sn 4(*l*)	2	3·602
	Sn 4(*i*)	2	3·507
	Sn 4(*j*)	1	3·651
Sn: 4(*f*)	Nb 8(*l*)	4	2·920
	Nb 4(*i*)	2	2·926
	Sn 4(*f*)	1	2·900
		1	2·760
	Sn 4(*j*)	2	3·763
Sn: 4(*i*)	Nb 8(*k*)	4	3·031
	Nb 8(*l*)	2	2·874
	Nb 4(*i*)	1	3·009
	Nb 4(*e*)	2	3·133
	Sn 8(*l*)	4	3·507
	Sn 4(*j*)	2	3·532
Sn: 4(*j*)	Nb 8(*k*)	4	2·798
	Nb 8(*l*)	2	2·916
	Nb 4(*i*)	2	2·966
	Sn 8(*l*)	2	3·651
	Sn 4(*f*)	2	3·763
	Sn 4(*i*)	2	3·532

The structure shows pronounced layering normal to the **a** direction. The layers at $x=0$ and $x=\frac{1}{4}$ are illustrated in Fig. 2. These layers are duplicated at $x=\frac{1}{2}$ and $x=\frac{3}{4}$ by the body-centering symmetry operation. The atoms on the $x=\frac{1}{4}$ and $x=\frac{3}{4}$ are not rigorously coplanar but deviations from coplanarity are small.

Discussion

The strongly developed linear chains of niobium atoms which run along the three axial directions in Nb_3Sn run only along the **a** direction in the Nb_6Sn_5 structure and then with a 5·5% increase in average bond length. A second difference between the two structures is that there are no Sn–Sn contacts in Nb_3Sn while in Nb_6Sn_5, Sn–Sn contacts as short as 2·76 Å occur. These short contacts alternate with contacts at 2·90 Å to form linear chains of tin which also run in the **a** direction.

Of the known intermetallic structures, the structure of Nb_6Sn_5 is probably most closely related to the Ti_6Sn_5 structure reported by Schubert, Frank, Gohle, Maldonado, Meissner, Raman & Rossteutscher (1963); such a relationship was foreseen by van Vucht, Bruning & Donkersloot (1963). It should also be noted that the chemical analyses and measured density (8·6 $g.cm^{-3}$) of these latter workers are in accord with the stoichiometry, Nb_6Sn_5, and the theoretical density, 8·72 $g.cm^{-3}$, of the present investigation. Layering in the Ti_6Sn_5 structure occurs normal to the c axis and coplanarity of the atoms within the layers is fixed by symmetry. The atomic arrangement in the $x=\frac{1}{4}$ layer of the Nb_6Sn_5 structure is closely analogous to the atomic arrangement of the $z=0$ layer of the Ti_6Sn_5 structure. The layer at $x=0$ of the Nb_6Sn_5 structure would also correspond to the layer at $z=\frac{1}{4}$ in the Ti_6Sn_5 structure if the mirror plane at $x=\frac{1}{2}$ in the Nb_6Sn_5 structure were replaced with a $c/2$ translation. While it is evident that there is a definite structural relationship between the Ti_6Sn_5 and Nb_6Sn_5, nonetheless the respective symmetries do generate atomic coordinations which differ between the two compounds.

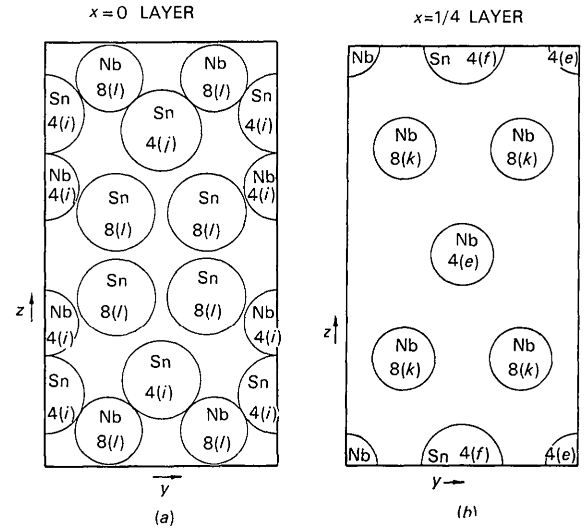

Fig. 2. Atomic arrangement on (a) the $x=0$ layer, (b) the $x=\frac{1}{4}$ layer, of Nb_6Sn_5.

Finally, the structure determinations indicate that in addition to Nb_3Sn the compounds Nb_6Sn_5 and $NbSn_2$ exist in the niobium–tin system. These two compounds have frequently been referred to as 'Nb_3Sn_2' and 'Nb_2Sn_3' in existing publications.

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