Letter

A new binary boride, Nb_5B_6

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The crystal structures of a number of binary borides are based on zigzag boron chains and double metal layers containing trigonal metal prisms with boron atoms in the centre of each prism (BMe_6 prisms). These structures are represented by five structure types; the MoB-, the CrB-, the V_5B_6-, the Ta_3B_4- and the V_2B_3-type structures [1]. The MoB-type structure is only represented by low temperature phases. Both the CrB- and the Ta_3B_4-type structures have several representatives among the transition metal borides. The first type is represented by VB, NbB, TaB, CrB, β-MoB and β-WB and the latter by V_3B_4, Nb_3B_4, Ta_3B_4, Cr_3B_4, Mn_3Bn_{34} and probably, though not yet well characterized, Ti_3B_4. For the V_5B_6- and the V_2B_3-type structures [2] only two representatives of each type have been reported. These are V_5B_6 and Ta_5B_6 [3, 4], and V_2B_3 and Cr_2B_3 [5, 6] respectively. Since all iso-stoichiometrical borides of the fifth group in the periodic system crystallize in the same structure type, it was decided to study the Nb–B system with respect to the occurrence of phases isomorphous to V_5B_6 and V_2B_3 and the Ta–B system with respect to the occurrence of a phase isomorphous to V_2B_3.

In the present work a new binary boride, Nb_5B_6, has been synthesized. The sample was prepared by arc-melting a pellet of pressed powder mixtures of the elements[a] under a purified argon atmosphere on a water-cooled copper hearth. X-ray examination of the sample was performed using a Guinier–Hägg focusing camera with Cu $K\alpha_1$ radiation ($\lambda = 1.540598$ Å) and with silicon ($a = 5.431065$ Å [7]) as internal calibration standard. The positions and the intensities of the reflexions were measured with a computer-controlled microdensitometer [8] and the unit cell dimensions were refined using the local least-squares program CELLKANT [9]. The result of this investigation showed that the sample consisted of NbB and Nb_5B_6. It was found that Nb_5B_6 is isomorphous with V_5B_6 and Ta_5B_6. It has an orthorhombic structure and belongs to space group *Cmmm*. The cell parameters obtained for Nb_5B_6 are $a = 22.768(2)$ Å, $b = 3.1539(3)$ Å and $c = 3.2992(3)$ Å (ESD:s in parentheses). Observed and calculated intensities are given in Table 1. The observed intensities are multiplied with a factor, a, to give

[a]Niobium, Highways International, Baarn, The Netherlands, claimed purity 99.9% and boron, H. C. Starck, Werk Goslar, F.R.G., analysed purity 99.4% with iron as main impurity.

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TABLE 1

Powder diffraction data of Nb_5B_6. Data are given for all observed reflexions. Non-observed reflexions with $I_{calc} \leq 2$ are excluded

h k l	d_{calc} (Å)	d_{obs} (Å)	I_{calc}	I_{obs}
6 0 0	3.7947	3.7973	6	7
0 0 1	3.2992	3.2985	3	vvw[a]
2 0 1	3.1688	3.1678	49	29
4 0 1	2.8544	2.8541	8	4
5 1 0	2.5927	2.5915	100	100
6 0 1	2.4898	2.4920	2	vvw[a]
10 0 0	2.2768	2.2766	28	16
1 1 1	2.2684	2.2690	10	19
3 1 1	2.1834	2.1830	100	160[b]
8 0 1	2.1550	2.1565	69	68
5 1 1	2.0386	2.0384	4	4
7 1 1	1.8669	1.8676	14	12
11 1 0	1.7305	1.7307	12	10
9 1 1	1.6936	1.6942	4	vvw[a]
0 0 2	1.6496	1.6498	20	37[b]
0 2 0	1.5770	1.5773	18	24
16 0 0	1.4230	1.4234	5	3
2 2 1	1.4118	1.4119	11	10
5 1 2	1.3918	1.3918	35	46
13 1 1	1.3889	1.3886	36	58
4 2 1	1.3803	1.3800	2	vvw[a]
15 1 0	1.3677	1.3677	9	5
10 0 2	1.3358	1.3358	13	9
10 2 0	1.2964	1.2962	12	11
8 2 1	1.2726	1.2727	33	36
11 1 2	1.1940	1.1941	10	7
18 0 1	1.1811	1.1808	17	9
0 2 2	1.1399	1.1403	19	36
2 0 3	1.0946	1.0946	4	w[c]

[a]Intensity too low to be determined by the atomic microdensitometer.
[b]Line overlap with NbB line.
[c]The background was too high to permit a good intensity determination.

intensities on the same scale as the calculated ones. The factor a is obtained from the formula $I_{calc} = aI_{obs} + b$, where b is the intercept on the I_{calc}-axis. Least-squares methods were used to calculate a and b but b was found to be negligible. Efforts to prepare single-phase samples of Nb_5B_6 by heat treatment or arc melting powder mixtures with different compositions have not been successful. An attempt was made to prepare Nb_2B_3 in the same way as Nb_5B_6. A number of heat treatments were also performed in the temperature range 1300–1700 °C. None of the samples, with an approximate composition of Nb_2B_3, contained anything but already known phases or Nb_5B_6. It has not been possible to synthesize either Na_5B_6 nor Ta_2B_3 with the method described above. However, single crystals of Ta_5B_6 have been prepared by a high temperature solution method [3].

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