The Crystal Structure of Cr_3B_4

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A number of borides isostructural with Ta_3B_4 have already been investigated by Andersson and Kiessling [1-3]. From his data on Mn_3B_4 Kiessling [4] concluded that there are boron-boron distances as short as 1.50 Å in this phase and he believed these short boron-boron distances to be present also in the other Me_3B_4 phases [3]. The value 1.5 Å is considerably shorter than any boron-boron distance previously reported. However, the atomic parameters are very uncertain. A reinvestigation of these compounds is therefore being undertaken in order to achieve greater accuracy. The present communication deals with the results obtained for the alloy Cr_3B_4.

Table 1. Atomic parameters in Cr_3B_4

Cr_I	in 2(c)	
Cr_{II}	in 4(*g*)	with $y = 0.1861 \pm 0.0000_6$
B_I	in 4(*g*)	with $y = 0.3607 \pm 0.0003$
B_{II}	in 4(*h*)	with $y = 0.4351 \pm 0.0003$

Experimental. The alloys were prepared by arc-melting mixtures of boron (99.0 %) and chromium (99.9 %) in an atmosphere of purified argon. X-Ray photographs were taken in a Guinier camera with Si as internal standard (aSi = 5.4306 Å) and with Cr$K\alpha$-radiation. The atomic parameters were determined from single crystal data, obtained in a Weissenberg camera with Mo$K\alpha$-radiation. The relative intensities were estimated visually using the multiple film technique and a calibrated intensity scale. The electron density projection $\varrho(xy)$, the structure factors and the scale- and temperature factors were computed and refined on the digital electronic computer BESK with programs available at BESK. The scattering factors according to Watson and Freeman [5] for chromium and Ibers [6] for boron were used.

Results. Within the limits of the experimental errors (estimated to be ± 0.04 %) no lattice parameter variations were observed. The lattice parameters of Cr_3B_4 obtained in this investigation (a = 2.986 Å, b = 13.02$_0$ Å, c = 2.952 Å) are in excellent agreement with Anderson's and Kiessling's [1] values (a = 2.984 Å, b = 13.02 Å, c = 2.953 Å).

The space group *Immm* derived by Kiessling in his study of Ta_3B_4 was confirmed. The atomic parameters obtained in this investigation for Cr_3B_4 are given in Table 1 with standard deviations calculated from Cruickshank's formula [7]. The final R-value for the 94 independent $hk0$ reflexions was 7.6 %.

Interatomic distances are given in Table 2. Of particular interest are the shortest boron-boron distances, which have a standard deviation of 0.02 Å. The difference between the two shortest non-equivalent boron-boron distances is not significant. Thus it has been shown that in Cr_3B_4 there exist no such abnormally short boron-boron distances as suggested by Kiessling [3].

Table 2. Interatomic distances in Cr_3B_4 shorter than 3 Å.

Cr_I	–	Cr_I	2.952	(2),	2.986	(2)	B_I	–	Cr_I	2.35	(2)
Cr_I	–	Cr_{II}	2.83$_7$	(4)			B_I	–	Cr_{II}	2.19	(4), 2.27 (1)
Cr_I	–	B_I	2.35	(4)			B_I	–	B_I	2.952	(2), 2.986 (2)
Cr_I	–	B_{II}	2.26	(8)			B_I	–	B_{II}	1.77	(2)
Cr_{II}	–	Cr_I	2.83$_7$	(2)			B_{II}	–	Cr_I	2.26	(4)
Cr_{II}	–	Cr_{II}	2.67$_6$	(4),	2.952	(2), 2.986 (2)	B_{II}	–	Cr_{II}	2.17	(2)
Cr_{II}	–	B_I	2.19	(4)	2.27	(1)	B_{II}	–	B_I	1.77	(2)
Cr_{II}	–	B_{II}	2.17	(2)			B_{II}	–	B_{II}	1.69	(1), 2.952 (2), 2.986 (2)

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1. Andersson, L.-H. and Kiessling, R. *Acta Chem. Scand.* **4** (1950) 160.
2. Kiessling, R. *Ibid.* **3** (1949) 90.
3. Kiessling, R. *Ibid.* **4** (1950) 209.
4. Kiessling, R. *Ibid.* **4** (1950) 146.
5. Watson, R. E. and Freeman, A. J. *Acta Cryst.* **14** (1961) 27.
6. Ibers, J. A. *Ibid.* **10** (1957) 86.
7. Cruickshank, D. W. J. *Ibid.* **2** (1949) 65.

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