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**The unit cell and space group of neopentyl alcohol.** By G. B. CARPENTER, *Metcalf Research Laboratory, Brown University, Providence, Rhode Island 02912, U.S.A.*

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Neopentyl alcohol crystallizes in space group  $Fm\bar{3}m$  with 4 molecules in a cell of edge  $a=8.83$  Å. The molecules are thus highly disordered about cubic close-packed centers.

Neopentyl alcohol,  $C(CH_3)_3CH_2OH$ , is of interest because its melting point of  $53^\circ$  is much higher than that of other five-carbon alcohols.

Single crystals were grown in capillaries of 0.2 mm internal diameter by slow cooling of melted samples. They were studied by Laue and precession methods with molybdenum radiation. The initial precession photographs showed a total of 14 reflections, which could be indexed on cubic axes as reflections of the two forms  $\{111\}$  and  $\{200\}$ . Measurements on the precession photographs recorded on Polaroid film according to the method of Swink & Carpenter (1967) gave a cell edge of  $a=8.83$  Å with a standard deviation of 0.02 Å. The standard deviation is so large because of the small number of measurable spots, all at low angles. More heavily exposed photographs revealed faint traces of reflections in the forms  $\{220\}$  and  $\{222\}$ .

The density measured by flotation is  $0.848$  g.cm $^{-3}$ , and the density calculated for four molecules in the unit cell is  $0.850$  g.cm $^{-3}$ .

The simplest interpretation of these observations is that the structure is face-centered cubic, space group  $Fm\bar{3}m$ , with four molecules centered at positions  $4(a)$ . Therefore each molecule is disordered so as to exhibit  $m\bar{3}m$  point symmetry. The structure thus appears to be the same cubic close-packed structure shown by neopentane (Mones & Post, 1952), carbon tetrachloride (Rudman & Post, 1966),

and other related substances, in the solid phase obtained from the melt.

With so few independent structure factors measurable, it seems unprofitable to try to learn more about the structure by X-ray diffraction at room temperature. Low temperature work on this substance is not now contemplated.

The infrared absorption spectrum was examined in an effort to determine the extent of hydrogen bonding in the highly disordered crystals. A sample of neopentyl alcohol was melted and then allowed to solidify between rocksalt windows, and the spectrum was recorded with a Perkin-Elmer model 621 spectrometer. An intense symmetrical band appeared in the O-H stretching region at about  $3340$  cm $^{-1}$  with a width at half height of approximately  $340$  cm $^{-1}$ ; this and the appearance of the rest of the spectrum indicate a moderate amount of hydrogen bonding.

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**The crystal structure of  $Fe_2AlB_2$ .** By WOLFGANG JEITSCHKO, *Department of Metallurgy, University of Illinois, Urbana, Illinois, U.S.A.*

(Received 7 June 1968)

$Fe_2AlB_2$  crystallizes in the orthorhombic space group  $Cmmm$  ( $D_{2h}^{19}$ ) with lattice constants  $a=2.9233 \pm 0.0010$ ,  $b=11.0337 \pm 0.0014$  and  $c=2.8703 \pm 0.0003$  Å. This compound was found to be isostructural with  $Mn_2AlB_2$  (Becher, Krogmann & Peisker, 1966, *Z. anorg. allg. Chem.* **344**, 140). The structure has been refined from single-crystal X-ray data, giving a final  $R$  value of 0.084 for the 140 independent structure factors observed. The structure is closely related to the  $CrB$ - and  $MoAlB$ -type structures.  $Fe_2AlB_2$  is either strongly paramagnetic or ferromagnetic at room temperature.

#### Introduction

In a publication on the ternary system iron-aluminum-boron Stadelmaier, Burgess & Davis (1966) reported the existence of a ternary phase close to the composition ' $Fe_3AlB_3$ '. From a structure-chemical viewpoint, aluminoborides are of interest because of the possible participation of aluminum atoms in building elements in the form of chains, nets, etc., which are formed by the boron atoms in borides (Aronsson, Lundström & Rundqvist, 1965). Although the structures of the various binary aluminumborides, as far as they are known (for references see Hoard &

Hughes, 1967), do not imply a covalent bond Al-B, this could not be ruled out for  $MoAlB$  (Jeitschko, 1966). However, the Al-B distance in this structure could not be determined accurately because the large scattering power of molybdenum prevented refinement of the free parameter of the boron atom.

#### Experimental

In their paper Stadelmaier, Burgess & Davis (1966) reported that the new ternary compound was found at a composition which is within the range of the primary crystallization of  $FeB$ . Primary crystallization of the ternary phase was, in turn,

observed by them in an unannealed alloy of composition  $\text{Fe}_{35}\text{Al}_{35}\text{B}_{30}$ . In the present work it was attempted to isolate single crystals of the new compound from an alloy of this composition, obtained by induction melting of the components in an alumina crucible under argon. The aluminum rich matrix of the sample was then dissolved in dilute hydrochloric acid, which attacked the crystals of the ternary phase at a considerably slower rate. During the subsequent structure determination it became apparent that the tentative composition 'Fe<sub>3</sub>AlB<sub>3</sub>' was incompatible with the size and symmetry of the unit cell. A quantitative flame emission analysis of the material gave the following results: Fe: 69.4+1.9 and Al: 16.3+0.5 both in wt.%; the balance was assumed to be boron. This analysis corresponds to  $\text{Fe}_{39.2}\text{Al}_{19.1}\text{B}_{41.7}$ . It supports the ideal composition  $\text{Fe}_2\text{AlB}_2$ , as deduced from the structure determination.

The single crystal used for the collection of the intensity data had the shape of an elongated rectangular prism, with the dimensions  $25 \times 28 \times 100 \mu$ . The crystal was mounted in a Weissenberg camera with the needle axis - which later turned out to be the [101] direction - perpendicular to the primary X-ray beam. The multiple-film technique (five films interleaved with Cu foils) was used, with  $\text{MoK}\alpha$  radiation. The intensities were estimated visually with the aid of an intensity strip. Absorption correction was made assuming a cylindrical crystal shape (*International Tables*, 1959).

#### Structure determination and refinement

The diffraction patterns could be indexed with an orthorhombic unit cell. Least-squares refinement, using back-reflection Debye-Scherrer data  $\lambda_{\text{CoK}\alpha_1} = 1.78892 \text{ \AA}$  resulted in the following lattice constants:

$$\begin{aligned} a &= 2.9233 \pm 0.0010 \text{ \AA}, & c &= 2.8703 \pm 0.0003 \text{ \AA}, \\ b &= 11.0337 \pm 0.0014 \text{ \AA}, & V &= 92.58 \pm 0.05 \text{ \AA}^3. \end{aligned}$$

The only systematic extinctions observed were those for a C-centered unit cell, leading to the diffraction symbol  $C^{***}$ . It was noted that the diffraction spots  $hkl$  were equal in intensity to those with  $h = \pm 2$  and/or  $l = \pm 2$ , apart from normal decline with higher diffraction angles. Thus, the structure has atoms arranged in rows parallel to [010] and separated from each other by  $\Delta x = \frac{1}{2}$  and  $\Delta z = \frac{1}{2}$ . Such an arrangement is also characteristic of the CrB and MoAlB structures. These also have similar unit-cell dimensions. Based on this information, a structure proposal with two formula units  $\text{Fe}_2\text{AlB}_2$  per unit cell was readily worked out by means of geometric considerations. The centrosymmetric space group  $Cmmm (D_{2h}^{10})$  was chosen for refinement by the least-squares method, using a full-matrix program by Gantzel, Sparks & Trueblood (1961). The function minimized was  $\sum w(\Delta F)^2$  with  $w = 1/F_{\text{obs}}^2$ . The values for the atomic scattering coefficients were taken from Table 3.3.1A of *International Tables* (1962) and they were corrected for the real part of the anomalous dispersion (Dauben & Templeton, 1955). Seven parameters were refined: the two positional  $y$  parameters, the three isotropic temperature factors for the atomic positions and two scale factors for the two reciprocal lattice planes evaluated. The refinement was considered to be finished when the changes of all parameters in the last two cycles were less than one tenth of the corresponding standard deviations. The final parameters are listed in Table 1. The final  $R$  value ( $R = \sum |F_o - F_c| / \sum F_o$ ) is 0.084 for the 140 observed structure factors, Table 2.

The agreement is good for high order  $h$  and  $l$  reflections as well. Therefore, it is not necessary to assume free  $x$  and  $z$  parameters, as allowed for by the noncentrosymmetric space groups  $C2mm (C_{2v}^2)$  and  $Cmm2 (C_{2v}^1)$ . The calculated density is  $5.75 \text{ g.cm}^{-3}$ .

Table 1. Atomic parameters of  $\text{Fe}_2\text{AlB}_2$  and their standard deviations

	Position	x	y	z	$B (\text{\AA}^2)$
Fe	4(j)	0	$0.35401 \pm 0.00015$	$\frac{1}{2}$	$0.51 \pm 0.02$
Al	2(a)	0	0	0	$0.49 \pm 0.05$
B	4(i)	0	$0.20714 \pm 0.00108$	0	$0.56 \pm 0.12$

#### Discussion

The  $\text{Fe}_2\text{AlB}_2$  structure is isotypic with  $\text{Mn}_2\text{AlB}_2$  (Becher, Krogmann & Peisker, 1966)\*. As can be seen from Fig. 1, the structure combines building elements of the CrB ( $B33$ ) and FeAl (CsCl-type) structures. It can be derived from the CrB structure by inserting one layer of aluminum atoms normal to the  $y$  direction between each pair of planes containing the  $[\text{Cr}_6\text{B}]$  prisms. With the insertion of another Al layer in a similar manner one arrives at the MoAlB structure.

\* These authors have described the structure in space group  $C222 (D_{6h}^2)$ . The special positions as required for the description of the  $\text{Mn}_2\text{AlB}_2$ -type structure are identical in the two space groups  $C222$  and  $Cmmm$ . However the total symmetry of the structure is that of  $Cmmm$  which is therefore given in the present paper.

Table 2. Observed and calculated structure factors for  $\text{Fe}_2\text{AlB}_2$

h	k	l	$F_o$	$F_c$	h	k	l	$F_o$	$F_c$	h	k	l	$F_o$	$F_c$	h	k	l	$F_o$	$F_c$
0	2	0	-	-16.6	3	9	3	<7.1	-2.1	3	7	2	27.4	-24.8	1	3	2	57.1	55.1
0	4	0	47.6	-47.3	3	11	3	10.8	-12.2	3	9	2	20.2	17.9	1	5	2	22.7	22.9
0	6	0	66.9	65.7	3	13	3	19.2	17.2	3	11	2	27.1	22.5	1	7	2	37.8	-35.9
0	8	0	32.6	38.1	3	15	3	8.9	10.7	3	13	2	14.5	-13.5	1	9	2	28.2	26.2
0	10	0	22.9	-28.0	3	17	3	15.1	-13.9	3	15	2	27.3	-1.4	1	11	2	34.5	31.4
0	12	0	46.3	4.7	3	19	3	6.5	5.6	3	17	2	21.2	17.6	1	13	2	18.2	-16.9
0	14	0	43.3	41.0	4	0	4	23.5	22.4	3	19	2	47.3	2.0	1	15	2	46.7	-1.1
0	16	0	<10.3	-9.1	4	2	4	47.6	-3.2	3	21	2	11.6	-10.6	1	17	2	25.1	21.9
0	18	0	<12.2	-11.3	4	4	4	8.4	-10.3	3	23	2	10.4	9.1	1	19	2	47.4	2.9
0	20	0	29.2	22.1	4	6	4	11.5	14.4	3	25	2	9.2	7.8	1	21	2	14.4	-12.7
0	22	0	<11.0	5.0	4	8	4	8.3	9.3	4	0	3	14.1	-13.7	1	23	2	12.9	11.0
0	24	0	11.4	-11.0	4	10	4	8.3	-10.0	4	2	3	47.1	7.1	1	25	2	9.7	9.4
0	26	0	<10.4	4.5	4	12	4	47.2	.9	4	4	3	23.6	22.6	1	27	2	7.6	-8.5
0	28	0	10.7	10.7	4	14	4	13.6	15.1	4	6	3	10.3	-9.8	1	29	2	20.4	-19.9
1	1	1	62.3	63.3	1	1	0	28.6	-28.7	4	8	3	7.7	-6.9	2	2	3	9.2	11.9
1	3	1	56.5	-53.2	1	3	0	72.2	81.9	4	10	3	26.9	21.8	2	4	3	34.1	35.3
1	5	1	12.1	13.6	1	5	0	31.9	31.9	4	12	3	47.4	.7	2	6	3	12.0	-13.5
1	7	1	64.5	58.6	1	7	0	47.6	-49.3	4	14	3	11.1	-10.2	2	8	3	7.5	-9.1
1	9	1	44.9	-2.7	1	9	0	32.2	33.9	4	16	3	7.5	8.5	2	10	3	35.6	31.6
1	11	1	21.2	-20.9	1	11	0	39.1	39.5	4	18	3	10.2	10.1	2	12	3	46.9	1.3
1	13	1	34.2	30.7	1	13	0	22.0	-19.9	4	20	3	46.3	-6.9	2	14	3	14.4	-12.9
1	15	1	20.3	18.6	1	15	0	46.2	-1.8	5	1	4	6.7	-5.6	2	16	3	10.3	11.5
1	17	1	22.9	-20.8	1	17	0	28.0	25.0	5	3	4	11.9	13.8	2	18	3	14.1	13.5
1	19	1	7.6	9.2	1	19	0	47.1	3.5	5	5	4	46.5	5.4	2	20	3	8.3	-8.8
1	21	1	21.9	17.4	1	21	0	14.5	-14.0	5	7	4	11.2	-11.9	2	22	3	46.6	-2.1
1	23	1	47.9	-6.5	1	23	0	14.0	12.2	5	9	4	5.7	7.6	2	24	3	16.3	13.3
1	25	1	47.3	-4.7	1	25	0	10.8	10.3	5	11	4	10.8	10.7	3	1	4	6.4	-7.6
1	27	1	12.0	10.8	1	27	0	10.3	-9.3	5	13	4	6.8	-7.1	3	3	4	20.5	20.7
1	29	1	52.3	58.6	2	0	1	36.4	-34.8	5	15	4	44.8	-1.3	3	5	4	7.3	8.9
2	2	2	7.1	-5.2	2	2	1	22.0	21.3	0	0	1	47.0	-53.0	3	7	4	17.4	-17.3
2	4	2	23.7	-22.1	2	4	1	56.9	57.5	0	2	1	29.3	30.1	3	9	4	9.3	11.7
2	6	2	34.4	35.1	2	6	1	21.2	-20.3	0	4	1	80.2	83.3	3	11	4	14.6	15.6
2	8	2	22.3	21.7	2	8	1	13.0	-12.5	0	6	1	30.5	-29.3	3	13	4	9.4	-10.1
2	10	2	20.9	-17.9	2	10	1	46.3	45.2	0	8	1	17.0	-16.4	3	15	4	47.1	-1.5
2	12	2	46.5	2.5	2	12	1	45.9	2.4	0	10	1	63.6	57.1	3	17	4	14.4	13.2
2	14	2	31.7	29.5	2	14	1	19.1	-16.0	0	12	1	45.3	3.4	3	19	4	46.2	1.2
2	16	2	47.3	-7.4	2	16	1	18.5	14.8	0	14	1	19.8	-18.4	3	21	4	7.3	-8.0
2	18	2	7.7	-9.3	2	18	1	20.4	16.9	0	16	1	22.1	17.2	4	0	5	8.8	-9.8
2	20	2	20.7	17.7	2	20	1	11.6	-10.4	0	18	1	22.2	19.1	4	2	5	46.6	4.6
2	22	2	47.3	4.1	2	22	1	47.3	-2.5	0	20	1	11.6	-11.2	4	4	5	13.8	14.5
2	24	2	8.5	-8.6	2	24	1	20.4	16.2	0	22	1	47.4	-2.8	4	6	5	6.9	-7.0
3	3	3	21.7	22.4	2	26	1	45.9	-2.1	0	24	1	24.3	17.8	4	8	5	46.3	-4.8
3	3	3	23.8	-20.4	3	1	2	10.8	-10.7	0	26	1	46.5	-2.3	4	10	5	14.7	14.4
3	3	3	46.7	5.6	3	3	2	32.1	33.1	0	28	1	6.8	-6.6	4	12	5	41.5	-5.6
3	3	3	25.9	24.6	3	5	2	14.1	14.7	1	1	2	17.5	-17.9	4	14	5	7.2	-7.3

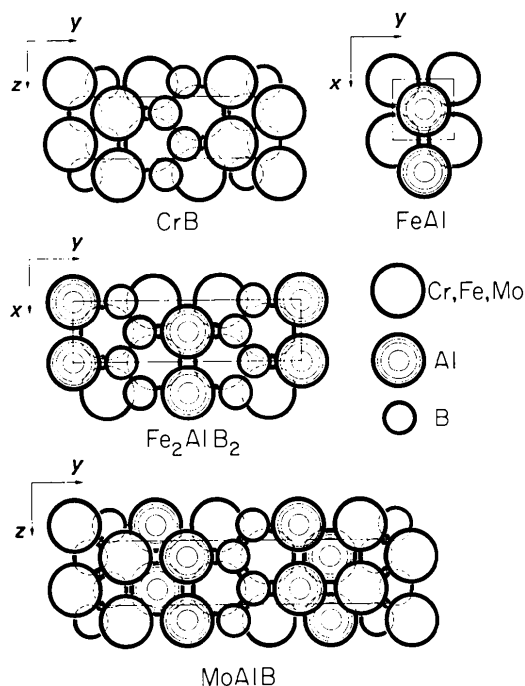


Fig. 1. Structure of  $\text{Fe}_2\text{AlB}_2$  as compared to the related structures of CrB, FeAl and MoAlB. Fully visible and partially visible atoms are separated by  $\frac{1}{2}$  translation period of the projection direction.

The interatomic distances (Table 3) Fe-Fe and Al-Al are longer than the sum of the coordination number 12 radii, whereas the distances Fe-Al are somewhat shorter. The boron atoms form zigzag chains with an angle of  $114.2 \pm 1.3^\circ$ . Although the distance Al-B is shortened, as compared to the Fe-B distances, it is considerably greater than the distance of about  $2.14 \text{ \AA}$  one would expect to find for a covalent Al-B bond. Thus the assumption of a covalently bonded B-Al net, with boron atoms forming  $sp^2$  and aluminum atoms  $sp$  hybrids, is not strongly supported by the observed interatomic distances.

$\text{Fe}_2\text{AlB}_2$  is either strongly paramagnetic or ferromagnetic at room temperature. Ferromagnetism has also been reported for  $\text{Mn}_2\text{AlB}_2$  (Becher, Krogmann & Peisker, 1966),  $\text{Fe}_2\text{B}$ , FeB and MnB (Cadeville & Meyer, 1962).

The author wishes to thank Professor P.A. Beck for many stimulating discussions and Miss E. Riemschneider for ex-

Table 3. Interatomic distances  $D$  ( $\text{\AA}$ ) in the structure of  $\text{Fe}_2\text{AlB}_2$

The values are compared with the sum of the atomic radii (C. N. 12 radii for Fe and Al; tetrahedral radius for boron).

		$D$	$r_A + r_B$	$\Delta$
Fe	2 Fe	$2.721 \pm 4$	2.52	+0.20
	2 Fe	$2.870 \pm 1$		+0.35
	2 Fe	$2.923 \pm 1$		+0.40
	1 Fe	$3.222 \pm 4$		+0.70
	4 Al	$2.606 \pm 2$	2.69	-0.08
Al	4 B	$2.156 \pm 5$	2.14	+0.02
	2 B	$2.165 \pm 10$		+0.02
	8 Fe	$2.606 \pm 2$	2.69	-0.08
	2 Al	$2.870 \pm 1$	2.86	+0.01
	2 Al	$2.923 \pm 1$		+0.06
B	2 B	$2.286 \pm 13$	2.31	-0.02
	4 Fe	$2.156 \pm 5$	2.14	+0.02
	2 Fe	$2.165 \pm 10$		+0.02
	1 Al	$2.286 \pm 13$	2.31	-0.02
	2 B	$1.741 \pm 14$	1.76	-0.02

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