work. One of the authors (B.N.D.) is grateful to C.S.I.R. (New Delhi) for partial support of the work.

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Acta Cryst. (1965). **18**, 574

Die Kristallstruktur des sauren Kaliummetasilikates $K_4(HSiO_3)_4$. Korrektur.

Von WALTRAUD HILMER, *Institut für anorganische Chemie der Deutschen Akademie der Wissenschaften zu Berlin, Berlin–Adlershof, Rudower Chaussee, Deutschland*

(*Eingegangen am* 16. *Oktober* 1964)

Auf Einwand von Frau Prof. Boll-Dornberger, im $K_4(HSiO_3)_4$ würde höhere Symmetrie als monoklin vorliegen, ergab ein Intensitätsvergleich von Aufnahmen verschiedener Kristalle, dass die Abweichungen der Intensitäten von rhombischer Symmetrie auf Absorption zurückzuführen sind. Intensitätssymmetrie, Auslöschungen und *I*-Statistik nach Howells, Phillips & Rogers der früher angegebenen Aufnahmen führen jetzt zur Raumgruppe *Bmab*. Dann ist $a_{\text{rhomb.}} = |\mathbf{a}-\mathbf{c}|_{\text{mon.}} = 11{,}50$ Å, $b_{\text{rhomb.}} = b_{\text{mon.}} = 11{,}25$ Å, $c_{\text{rhomb.}} = |\mathbf{a}+\mathbf{c}|_{\text{mon.}} = 9{,}65$ Å; $Z = 16$. Berücksichtigung der höheren Symmetrie führt zu folgenden Koordinaten in der rhombischen Zelle:

	x/a	*y/b*	*z/c*
K(1)	250	250	737
K(2)	0	043	822
Si(1)	500	184	986
Si(2)	302	0	0
O(1)	0	310	560
O(2) (=O(2, 3) mon.)	389	110	044
O(4)	500	199	825
O(5) (=O(5, 6) mon.)	227	029	870

Die in der Veröffentlichung angegebenen *R*-Faktoren ändern sich von 13,1% auf 12,8% und von 9,5% auf 9,3% (bzw. 17,6% auf 17,8%). Ein zur Kontrolle berechneter Bereich von (1*kl*)-Reflexen führte ebenfalls zu guter Übereinstimmung zwischen F_o und F_c.

Die Si–O-Abstände ändern sich im Mittel um 0,01 Å (maximal um 0,03 Å), $\sphericalangle$Si(1)–O(2,3)–Si(2) = 140,7°.

Ich danke Frau Prof. Boll-Dornberger für ihren Hinweis.

Acta Cryst. (1965). **18**, 574

Crystal data on $FeAlO_3$.

By R. R. DAYAL, J. A. GARD and F. P. GLASSER, *Department of Chemistry, University of Aberdeen, Scotland*

(*Received* 21 *September* 1964)

The $FeAlO_3$ phase was discovered by Richardson, Ball & Rigby (1954), who synthesized it by heating a 1:1 Fe_2O_3–Al_2O_3 mixture in oxygen at 1400° C. They also established that the product was a homogeneous single phase, distinct from α-Al_2O_3, α-Fe_2O_3 or α-$(Al, Fe)_2O_3$ solid solutions. This $FeAlO_3$ phase has subsequently been encountered by others, principally Atlas & Sumida (1958), Muan & Gee (1955) and Muan (1958). The latter workers explored the small range of stoichiometry and the limited range of thermodynamic stability of this phase; for example at $P_{O_2} = 1$ atm. the stability range is 1318°–1495 °C. The present authors encountered the $FeAlO_3$ compound during equilibrium studies in the system CaO–Al_2O_3–Fe_2O_3. Powder X-ray data were obtained from nearly pure preparations; these data (Table 1 (*c*)) agree very closely with those of Richardson *et al.* (1954), rather less well with those of Atlas (1958). Richardson *et al.* obtained a unit cell from trial-and-error procedures; the correctness of the cell was apparently supported by an analogy between $FeAlO_3$ and that of κ-Al_2O_3. Present efforts to improve the fit between d_o and d_c were, however, unsuccessful. Efforts to grow single crystals of $FeAlO_3$ would obviously be handicapped by the limited stability of this phase; the prospect of growing single crystals appeared uninviting. It was therefore decided to examine the relatively fine-grained powders by selected-area electron diffraction.

A 1:1 mixture of coprecipitated hydrous oxides of Fe^{3+} and Al^{3+} was prepared. Gentle ignition at *ca.* 500 °C produced a nearly amorphous starting material. Heating at 1370° ± 3 °C for four days in air gave a nearly pure $FeAlO_3$ preparation, judging by the near absence of α-Al_2O_3 and α-Fe_2O_3 reflexions in powder photographs. Fe_3O_4 (magnetite) was absent. The sample adhered strongly to a small permanent magnet.

Samples were crushed gently and dispersed in isopropyl alcohol using ultrasonic vibration. Drops of this suspension were transferred to specimen grids carrying carbon films lightly coated with evaporated aluminum for calibration of the camera constant λL. The specimens were examined with an A.E.I. type EM3 electron microscope equipped with a goniometric stage permitting rotation and a maximum tilt of 45°, modified from a design of Burge & Munden (1960).

Approximately 30 crystals were examined. These all

Table 1. *Crystallographic and physical data for* $FeAlO_3$ *and related phases*

(*a*) Electron diffraction data
Unit cell: Orthorhombic, $a=8{\cdot}60$, $b=9{\cdot}25$, $c=5{\cdot}0$ Å; $\alpha=\beta=\gamma=90°$. Systematic absences: $0kl$, $l=2n$; $hk0$, $h+k=2n$; $(h00)$, $h=2n$; $(0k0)$, $k=2n$; $(00l)$, $l=2n$. Probable space group $Pc2_1n$ or $Pcmn$

(*b*) Density and cell content
Density (calculated from methylene iodide displacement, 27 °C), 4·41–4·45
Cell contents, $Fe_8Al_8O_{24}$

(*c*) X-ray powder data. Indices based on cell with $a=8{\cdot}60$, $b=9{\cdot}25$, $c=4{\cdot}97$ Å. Powder data obtained both on film and with diffractometer. *d*-Spacings were obtained with a Philips Type PW 1051 diffractometer, Co $K\alpha$ radiation. Diffractometer standardized with Si standard

Richardson *et al.* (1954) *d* (Å)	Present studies *I*	*d* (Å)	*I*	*hkl*	d_c (Å)
6·30	*m*	6·30	*m*	110	6·30
4·69	*vw*	4·63	*w*	020	4·62
		4·2	*vvw*	101	4·33
		4·3		200	4·30
3·90	*w*	3·91	*mw*	111	3·92
		3·66	*vvw*	?*	—
		3·49	*vvw*	?†	—
		$3{\cdot}24_5$	*vw*	201	3·26
3·14	*s*	3·145	*ms*	121	3·16
				220	3·15
		3·073	*vvw*	211	3·08
2·90	*s*	2·898	*ms*	130	2·90
2·69	*vs*	2·657	*vs*	221	2·67
		2·572	*vvw*	?‡	
2·50	*m*	2·499	*m*	131	2·51
				002	2·49
				301	2·485
2·39	*m*	2·391	*ms*	012	2·40
				311	2·40
				102	2·39
2·33	*vw*	2·32	*vw*	112	2·32
				040	2·31
2·23	*w*	2·234	*vw*	231	2·24
2·18	*s*	2·180	*s*	022	2·20
				321	2·19
2·12	*m*	2·120	*mw*	122	2·13
				212	2·10
				330	2·10
2·02	*vw*	2·02	*vvw*	141	2·04
				240	2·04
1·95	*vw*	1·935	*ms*	222	1·96
				420	1·95
				411	1·94
				331	
				032	
		1·889	*w*	132	1·88, 1·89
				241	
				302	

(*d*) Comparison of $FeAlO_3$ and $GaFeO_3$ phases

	$FeAlO_3$	$GaFeO_3$ (Wood, 1960)
a	8·60 Å	8·75 Å
b	9·25	9·40
c	4·97	5·07
	Orthorhombic Pseudohexagonal	Orthorhombic Pseudohexagonal
Space group	$Pc2_1n$ or $Pcmn$	$Pc2_1n$
Z	8	8
V	395 Å^3	417 Å^3

* α-$(Fe,Al)_2O_3$ reflexion. For pure Fe_2O_3, $d=3{\cdot}66$ Å, $I=ms$.
† α-$(Al,Fe)_2O_3$ reflexion. For pure Al_2O_3, $d=3{\cdot}49$ Å, $I=vs$.
‡ α-$(Al,Fe)_2O_3$ reflexion. For pure Al_2O_3, $d=2{\cdot}552$ Å, $I=vs$.

had irregular shapes. The $0kl$ and $h0l$ sections of the reciprocal lattice were readily examined. However only one crystal could be tilted to record the $hk0$ zone, and this crystal required *ca.* 45° of tilt. This apparent preferred orientation is probably due to cleavage. All the patterns could be indexed on the unit cell shown in Table 1 (*a*). The systematic absences were found from comparison of adjacent Laue zones (see, *e. g.* Gard, 1956; 1964).

These cell dimensions and systematic absences indicate that the compound is isostructural with $GaFeO_3$ (compare Table 1 (*d*)).

α-Al_2O_3 and α-Fe_2O_3 (corundum type) contain layers of approximate close packing. The ideal density of a hypothetical corundum compound having the composition $FeAlO_3$ would be given by $\frac{1}{2}(d_{\alpha\text{-}Al_2O_3}+d_{\alpha\text{-}Fe_2O_3}) = \frac{1}{2}(3{\cdot}98+5{\cdot}25) = 4{\cdot}62$ g.cm^{-3}. This density is sufficiently close to that found for $FeAlO_3$ (4·45) to suspect that it also contains oxygen atoms in approximate close packing. Moreover, in the $h0l$ zone all reflexions $h=3(2n+1)$ are systematically strong, indicating a hexagonal subcell with $c=2{\cdot}87$ Å ($=a/3=c/\sqrt{3}$): the cell dimensions suggest four hexagonally packed layers of oxygens parallel to (010).

Efforts to determine the $FeAlO_3$ structure will be postponed until the nearly complete results of the $GaFeO_3$ structure (Abrahams, personal communication) are available. $FeAlO_3$ is not, however, isomorphous with κ-alumina, judging from the hexagonal cell dimensions given by Brindley (1961) for the two κ-like phases: $a=9{\cdot}70$ Å, $c=$?, and $a=16{\cdot}78$ Å, $c=$?

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