order for hydrogen bond formation. This Cl–O(2') distance is shorter than the normal value of 3.2 Å and indicates that the bond is comparatively strong. The non-bonding interatomic distances less than 4 Å are Cl–C(2), 3.867 Å; Cl–C(3)', 3.837 Å; Cl–C(4'), 3.572 Å; and Cl–C(5'), 3.563 Å. The observed distances are all larger than the sum of the radii of the corresponding atoms. It is seen from Fig.3 that the molecules are packed in such a way that the non-polar groups face each other across planes and similarly for the polar groups.

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The Crystal Structure of CeFe7, PrFe7, NdFe7, and SmFe7*

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The isostructural phases CeFe₇, PrFe₇, NdFe₇, and SmFe₇ are rhombohedral; the probable space group is $R\overline{3}m$. The lattice constants for the triply primitive hexagonal unit cells are:

	а	С	c/a
CeFe ₇	8·488±0·003 Å	12·402±0·007 Å	1.461
PrFe ₇	8.582 ± 0.003	12.462 ± 0.007	1.452
NdFe7	8.578 ± 0.003	12.462 ± 0.007	1.453
SmFe ₁₇	$8{\cdot}554\pm0{\cdot}003$	$12{\cdot}441\pm0{\cdot}007$	1.454

The hexagonal unit cells contain seven rare earth and forty-nine iron atoms. The atomic arrangements are intermediate between those of the D2a-CaCu₅ and Th₂Zn₁₇ structural types. A statistical occupancy of one set of positions has been proposed to account for the observed rhombohedral symmetry.

Introduction

Nassau, Cherry & Wallace (1960) have reported a phase COITESPONDING to the formula CeFe₅ as having the $D2_a$ -CaCu₅ structure. Jepson & Duwez (1955) also reported CeFe₅ but observed that only 32 of 39 lines on their powder pattern could be accounted for by the CaCu₅ structure and that the intensity match was poor. Zarechnyuk & Kripyakevich (1963) reported the phase Ce₂Fe₁₇ (a=8.47, c=12.41 Å) to be isostructural with Th₂Zn₁₇ (Markarov & Vinogradov, 1956). Kripyakevich, Terekhova, Zarechnyuk & Burov (1963) reported a Nd-Fe phase, in equilibrium with α -Fe, to be isostructural with Th₂Zn₁₇. Savitskii, Terekhova, Burov, Naumkin & Markova (1965) have indicated that this Nd-Fe phase corresponds to the composition Nd₂Fe₁₇ (a=8.59, c=12.47 Å). Kripyakevich & Frankevich (1966) have recently reported the phase Pr₂Fe₁₇ (a=8.58, c=12.47 Å) to have the Th₂Zn₁₇ structure.

Ray, Strnat & Feldmann (1964) have reported the preparation and magnetic properties of isostructural intermediate phases having the approximate compositions RFe₇ (R=Ce, Pr, Nd). They have shown these RFe₇ phases to form peritectically and that each represents the most iron-rich intermediate phase existing in its alloy system. The powder X-ray diffraction patterns of the RFe₇ phases have been indexed on the

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basis of hexagonal unit cells. Systematic extinctions observed on the diffraction patterns indicate rhombohedral symmetry, although an ordered rhombohedral structure is not consistent with the densities, compositions, and unit-cell volumes observed for the RFe7 phases. Preliminary efforts to resolve the apparent structure-composition inconsistency were hampered by the lack of suitable single crystal X-ray data and reliable compositional analyses.

In the present investigation, X-ray data obtained from an untwinned PrFe₇ crystal plus confirmation of the compositions of the RFe₇ phases by electron microprobe analyses have been employed to determine the atomic arrangements in PrFe₇ and by inference the structures of CeFe₇, NdFe₇, and a phase not previously reported, SmFe₇.

Experimental

Electron microprobe analyses of Ce-Fe, Pr-Fe, and Nd-Fe alloys are summarized in Table 1. The analyses were performed by the Advanced Metals Research Corporation, Somerville, Massachusetts. These results confirm the compositional formula RFe7 originally assigned to the Fe-rich phases in the Ce-Fe, Pr-Fe, and Nd-Fe alloy systems by metallographic and X-ray diffraction analyses. Chemical analyses have not been obtained for SmFe₇. The inclusion of SmFe₇ is based upon the similarity of its powder pattern to the other RFe₇ phases and upon metallographic evidence obtained from homogenized Sm-Fe alloys. A Sm-Fe alloy containing 87.5 at.% Fe was observed to be single phase after homogenization for 72 hours at 800°C.

Unit-cell constants were obtained from Debye-Scherrer powder patterns employing chromium $K\alpha$ radiation. The results for the triply primitive hexagonal unit cells of the RFe7 phases are given in Table 2. Values for a_0 were obtained by extrapolating hk0 data versus the function of Nelson & Riley (1945), while c_0 values were obtained from *hkl* reflections with large l indices by the graphical method of successive approximations of Lipson & Wilson (1941). The powder patterns for CeFe₇, PrFe₇, NdFe₇, and SmFe₇ are given in Table 3. A double beam, recording microdensitometer was employed to obtain the line intensities. Line intensities listed for PrFe₇ and NdFe₇ were obtained by graphical integration while those for CeFe7 and

Table 2. Lattice constants for the triply primitive hexagonal unit cells of the RFe₇ phases

Phase	<i>a</i> _o	c_0	c/a
CeFe ₇	8·488 ± 3 Å	12.402 ± 7 Å	1·461
PrFe ₇	8·582 ± 3	12.462 ± 7	1·452
NdFe ₇	8·578 ± 3	12.462 ± 7	1·453
SmFe ₇	8.578 ± 3 8.554 ± 3	12.402 ± 7 12.441 ± 7	1.454

SmFe₇ were approximated by multiplying the peak heights times the peak widths at half maximum.

Several fragments obtained by crushing single-phase PrFe₇ and NdFe₇ alloys were inspected to find a crystal suitable for single-crystal analyses. Invariably, these fragments were found to be twinned, and reflections having -h+k+l=3n and h-k+l=3n were observed simultaneously on the Weissenberg diffraction patterns. It is suspected that much of the twinning was produced by crushing the alloys. Extensive twinning was also noted in the microstructures of the annealed alloys.

A many faceted PrFe7 crystal, approximating a sphere with an average diameter of 0.2 mm, was isolated from a Pr-Fe alloy containing 83 at.% Fe. Weissenberg patterns of this crystal displayed no evidence of twinning. The alloy had been previously annealed at 800°C for 40 hours, then allowed to remain in an unsealed container for several months. Immediately after annealing, the alloy was composed of PrFe₇ crystals in a eutectic matrix of Pr plus another Pr-Fe phase. On exposure to the atmosphere, the eutectic matrix of the alloy oxidized to a pale green powder, leaving many PrFe₇ crystals free of the matrix and free of mechanical distortion.

Intensity data were obtained from the PrFe₇ crystal by visually comparing the diffraction spots on timed Weissenberg photographs with a set of standard diffraction spots obtained from the same crystal. The intensities of 66 out of a possible 76 independent planes within the sphere of reflection for Cr $K\alpha$ radiation were observed and measured.

Structure determination

Four structural types corresponding to the composition A_2B_{17} have been reported: Th_2Ni_{17} and Th_2Fe_{17} by Florio, Baenziger & Rundle (1956) and Th₂Zn₁₇ and U₂Zn₁₇ by Markarov & Vinogradov (1956). All of these

Alloy	Microstructure	Fe	RE
Arc melted 87.5 Fe-12.5 Pr alloy used as standard. Annealed 87.5 Fe-	Peritectic average center of dendrite peritectic phase Single phase	$\begin{array}{r} 87.5 \\ 100 \pm 0.5 \\ 87.6 \pm 0.2 \\ 87.3 \pm 0.2 \end{array}$	$12.5 (Pr) 0.15 \pm 0.03 12.4 \pm 0.2 12.7 \pm 0.2 (Pr)$
12.5 Pr alloy. Annealed 87.5 Fe- 12.5 Nd alloy.	Single phase	87.4 ± 0.2	12.6 ± 0.2 (Nd)
Annealed 87.5 Fe- 12.5 Ce alloy.	Single phase	87·4±0·2	12.0±0.2 (Ce)

Table 1. Electron microprobe analyses of rare earth-iron alloys

Compositions in at. %

 A_2B_{17} structures are derived from the $D2_d$ -CaCu₅ structure by the substitution of two of the smaller B atoms for one A atom at one-third of the A positions in AB_5 as shown in Fig.1. The A_2B_{17} structures differ from one another in the manner in which the substituted A positions are arranged. Th₂Ni₁₇ is hexagonal with the substitution occurring in a BC stacking sequence [Fig. 2(a)]. Th₂Zn₁₇ [Fig. 2(b)] is rhombohedral with an ABC stacking sequence for the substituted A positions. Th_2Fe_{17} [Fig. 2(c)] has a more complex ABC stacking sequence which reduces the symmetry to monoclinic. On the basis of powder X-ray data, Markarov & Vinogradov reported U_2Zn_{17} to be hexagonal with an ABCACB stacking sequence. More recently, however, Vold & Peterson (1961) have shown by single-crystal analyses that U_2Zn_{17} is not a distinct A_2B_{17} structural type, but rather is dimorphic, one form having the Th_2Ni_{17} structure and the other the Th_2Zn_{17} structure, both of which can be retained at room temperature by furnace cooling.

Ray, Strnat & Feldmann (1964) have suggested that the crystal structures of the RFe_7 are intermediate be-

tween the CaCu₅ and Th₂Zn₁₇ arrangements. The measured density of $PrFe_7$ is 7.73 ± 0.10 g.cm⁻³ while the calculated density of the triply primitive PrFe₇ unit cell containing 7 Pr and 49 Fe atoms is 7.78 g.cm⁻³. It was observed that both the density and composition of PrFe₇ could be accounted for by assuming a structure composed of one AB₅ layer alternating with two A_2B_{17} layers, *i.e.* $A_3B_{15} + 2A_2B_{17} = A_7B_{49}$. An ordered arrangement of this type, however, is not consistent with the observed rhombohedral symmetry. To account for the rhombohedral symmetry, $\frac{1}{4}$ Pr and $\frac{4}{5}$ Fe atoms were statistically assigned to each of the substituted A positions, and these were then arranged in a Th₂Zn₁₇ layering sequence, as shown in Fig. 2(d). The initial positions for this arrangement are given in Table 4(a). For this trial structure, the reliability index $\Sigma |(F_o - F_c)|/$ $\Sigma |F_o|$ was 0.29. A reduction in the reliability index to 0.19 (including unobserved reflections) was obtained by shifting the atoms from their ' Th_2Zn_{17} ' positions to those found by Zalkin, Sands & Krikorian (1959) for Nb₂Be₁₇, given in Table 4(b). The positional parameters assumed for PrFe₇ are also in close agreement

Table 3. Powder diffraction patterns for CeFe7, PrFe7, NdFe7, and SmFe7

Chromium $K\alpha$ radiation,	hexagonal	indexing,	space	group	R3m
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								-			•	•					
	Cel	e,	PrFe	7	NdF	'e7	Sm	Fe7		Cel	e,	PrFe	7	NdI	e,	Sm	Fe7
hk/	d, Å	Io	d, Å	Io	d, X	Io	d, 🎗	I _o	hkt	d, Å	I _o	d, Å	Io	d, X	I _o	d, X	Io
011	6 . 296	z	(6.381)						045			(1 4892)					
012			(4.774)						051			1. 4753	2				
110	4.220	5	(4.289)		4.280	2	4. 261	5	052			1.4452	2				
003			(4.154)		4.134	2	4.114	5	028			(1.4365)					
021	3.520	5	3.559	8	3.554	5	3.541	10	330	1.4140	5	1.4301	14	1.4299	17	1.4250) 10
022	3.156	5	3.193	3	3.184	8	3.176	10	235	1.3945	5	1.4067	14	1.4065	7	1.4022	215
113	Z.960	40	2.981	36	2.978	39	2.969	40	241			(1.3950)					
014	2.855	10	2.870	13	2.868	8	2.858	20	009			(1. 3846)					
12Ī	2.709	5	2.738	9	2.735	8	2.725	10	242			(1.3695)					
122	2. 535	5	2.559	8	2.556	5	2.548	10	128			1.3622	2				
030	2.449	50	2.476	37	2.472	36	2.465	40	333]	1 2202		1.3525	45	1.3516) 1	1 2475	
024	2.370	40	2.386	20	2.384	18	2.377	30	1.7- 1	1.3382	60	1 2470	• •	1 140 1	}55 J	1.34/5	, 60
015			(2.363)						137 9	1 22/2	-	1.3470	14	1. 3492),	1 22/1	
220	2.121	80	2.145	75	2.141	65	2.135	60	054 ເຕັ ໄ	1. 3202	5	1. 3410	9	1. 3410	15	1.3305	· 5
033	2.107	90	2.126	82	2.124	68	2.118	60	151 }	1.3118	10	1. 3268	12	1.3200	17	1. 3229	10
124	2.081	10	2.086	17	2.085	16	٦		119 J			1.3172	12	1.3174	18	1.3148	10
006	2.068	60 L	2 075	61	1 2 075	-0	2.071	60d	152	1.2907	15	1.3050	8	1.3043	7	1.3000	5
025	2.057	10 ∫	2.015	04	ſ 2.075	59	J		047			1.2851	1			1.2844	F 5
131			2.033	3					244			ר 2801 1.		1.2799			
132			1.956	3					146	1.2672	30	1.2780	47	1.2778	47	1.2763	30
223	1.887	60	1.906	33	1.904	33	1.898	• •	055 J			1.2762		1.2761	,		
116	1.859	10 L	1 867	12	1 866	20	1 061	204	138			1.2425	2	1			
125	1 850	10 1	1.001	12	f 1.000	20	J 1.001	200	060	1.2251	45	1.2386	67	1.2380	72	1.2345	i 60
041	1 817	5	1 838	5	1 8 3 6	10	1 0 7 0	10	237	1.2218	30	1.2315	60	1.2310	47	1.2282	30
042	1. 762	5	1.050	3	1.050	4	1.027	10	0,1,10			(1.2290)					
017	1 221	-	1. 701	,	1	-	1. 772	10	154	1.2146	30	1.2265	48	1.2264	44	1.2230	30
017	1.725	5	1. 731	4	1. 731	8	1.727	10	245	1.2122	15	1.2235	40	1. 2229	20	1 2202	> 20
134	1.703	5	1.719	3	1.717	6	1.712	10	341			1.2157	8	1. 21 54	5	1 21 27	75
231	1.672	5	1.689	1	1.688	5	1.682	10	039	1.2013	100	1.2087	100	1.2085	100	1.2063	3 100
232	1.632	5	1.643	14	1.641	26	1.638	10	342			1.1989	15	1 1984	14		
140			1.622	3	1.621	7	1.615	10	048	1,1852	15	1.1937	17	1,1935	22	1 1000	215
027	1.596	5	1.606	5	1.606	8	1.602	10	250			1.1895	6	1 1896	4		, 15
044			(1.595)						063			1.1869	8	1.1865	. q		
036	1.580	10	1.592	8	1.591	12	1.587	10	0.2.10	1,1751	15	1,1814	28	1, 1813	30	1 1 794	4 1 5
135			(1.588)						336	/		1.1784		1.1782	່້າ		
018			(1.524)						· · · · }	1.1655	15		► 30		`}30 }	1.1731	115
143	1.495	50	1.511	33	1.510	29	1.506	40	155 J			1.1765		1.1762	9		
127 -		-	1.5036	29	1, 5030) 22	1.4994	20	229	1.1562	15	1.1634	33	1.1633	333	1.1611	115
234			1.4950]		1.4950).)		238			1.1500	11	1.1498	313		
226 -	1.4813	5 60	1.4919	65	1.4918	} 63	} 1.489	60									

with those found by Vold & Peterson for the rhombohedral modification of U_2Zn_{17} . Least-squares structure refinements were employed in both of the previous investigations.



Fig. 1. Comparison of the layers in (a) $D2_{d}$ -AB₅ and (b) A₂B₁₇ structures. Alternate layers (c) are common to both types. The dashed lines in (a) and (c) indicate the unit-cell edges for AB₅ structures. The two B atoms in (b) marked + and - lie above and below and are tangent to the plane of atoms and to each other.

Table 5. Observed and calculated structure factors for PrFe₇, hexagonal indexing

	ucn	15 101	· · · · /,		ing i			5
hk	Fo	Fc	hk	Fo	Fc	<u>hk/</u>	<u>F</u> _0	Fc
003	11	14	048	23	23	146	9	1
006	111	107	051	10	-11	151	12	-12
009	14	19	052	10	-17	152	13	-15
011	< 5	.1	054	15	-26	1 54	24	- 24
012	< 5	1	055	13	-13	155	17	-15
014	25	-14	060	76	80	2 2 0	75	75
015	6	- 6	063	10	- 2	223	36	- 33
017	22	-14	110	< 5	7	226	50	52
018	< 5	4	113	30	33	229	15	-17
01,10	< 5	- 2	116	11	- 6	231	13	-17
021	20	-12	119	18	30	232	29	-13
0 2 2	12	-12	121	14	- 8	234	23	-23
024	42	- 32	122	12	-12	235	25	- 20
025	31	- 24	124	27	-23	237	11	- 25
027	22	-15	125	14	-11	238	6	-7
028	< 5	4	127	31	-19	241	6	- 5
02,10	24	- 21	128	6	- 5	242	5	- 5
0 30	54	54	1 31	< 5	4	244	23	- 21
033	68	74	1 3 2	6	4	245	29	-15
036	18	33	134	10	-7	250	10	10
039	60	62	1 3 5	< 5	-,5	3 3 0	36	36
041	20	15	137	13	- 8	333	41	53
042	15	15	1 3 8	5	7	336	21	23
044	< 5	-4	140	9	12	341	12	15
045	< 5	2	143	23	31	342	14	10
047	7	7						

A ATOMS

Table 4. Hexagonal atomic parameters in $PrFe_7$, space group $R\overline{3}m$ (i) Initial positions based on Th_2Zn_{17} . (ii) Refined positions based on Nb_2Be_{17} .

	(i)			(ii)*				
	х	у	Z	x	у	Z		
1 Pr_1 in (a) (statistically)	0	0	0	0	0	0		
4 Fer in (c) (statistically)	0	0	0.1	0	0	0.103		
6 Pr $_{\rm II}$ in (c)	0	0	ł	0	0	0.340		
9 Fe ₁₁ in (d)	ł	0	12	0-500	0	0.200		
18 Ferry in (f)	Ĩ	0	Ō	0.297	0	0		
18 Fe_{IV} in (h)	16	5	$\frac{1}{2}$	0.164	0.836	0.494		

* The parameters in (ii) have been shifted by 0,0,1 from those originally listed for Nb2Be17 (Zalkin, Sands & Krikorian, 1959).



Fig. 2. Comparison of the layering arrangements in (a) Th₂Ni₁₇, (b) Th₂Zn₁₇ (c) Th₂Fe₁₇, and (d) PrFe₇

The observed and calculated structure factors for $PrFe_7$ are given in Table 5. Atomic scattering factors listed in *International Tables for X-ray Crystallography* (1962) were employed for the structure factor calculations. Absorption and temperature factor corrections have been neglected. Interatomic distances based on the assumed atomic positions in $PrFe_7$ are given in Table 6. The only distances which appear to be abnormally short are those between the statistically occupied Pr_I and the Fe_{III} atomic positions.

Table 6.	Interatomic	distances	in	PrFe
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$Pr_{I} - 2Pr_{II}$	4.24	Fe _{III} –1Pr _I	2.55
6Fe11	3.24	2Pr11	3.03
6FeIII	2.55	2Fe _I	2.85
6Fe _{IV}	3.12	2Fe11	2.46
		2Fem	2.63
Pr11-1Pr1	4.24	1Fem	3.48
1Pru	3.99	2Ferv	2.59
1Fer	2.95	2Ferv	2.66
3Ferr	3.28		
6Fem	3.03	Ferv-1Pr	3.12
3Fe _{IV}	3.27	1Pr1	3.27
3Fe _{1v}	3.20	1 Pr 11	3.20
3Ferv	3.10	1Pru	3.10
		1Fer	2.66
Fe ₁ -1Pr ₁₁	2.95	2Ferr	2.50
1Fe ₁	2.57	2Ferr	2.59
3Ferr	2.60	2Fem	2.66
6Fem	2.85	2Ferv	2.44
3Fe _{IV}	2 .66		- · ·
Fe11-1Pr1	3.24		
2Pru	3.28		
2Fer	2.60		
4Fem	2.46		
4Fe _{IV}	2.50		

Comparison of the powder diffraction pattern of $PrFe_7$ with those of $NdFe_7$ and $SmFe_7$ indicates no significant atomic shifts would be expected for these phases. The larger c/a ratio of $CeFe_7$ alters its pattern slightly and may indicate a slight adjustment of some of the atomic positions from the $PrFe_7$ arrangement.

Discussion

Intermediate phases which have some atomic positions statistically occupied are generally found to be stable over a range of compositions. No evidence of a solubility range near 87.5 at.% Fe has been observed in either Pr-Fe or Nd-Fe alloys homogenized for long periods between 800 and 1000°C. However, information from metallographic and X-ray diffraction studies (Ray, 1965) and from magnetic measurements (Strnat, 1965) of Ce-Fe alloys containing from 83 to 92 at.% Fe and homogenized at 837°C for 750 hours indicates that the single-phase field in this system may extend from 84.5 to 88.5 at.% Fe. If these indications concerning Ce–Fe alloys are correct, they would account for the CeFe₅ composition (83.3 at.% Fe) reported by Jepson & Dewez (1955) and for the Ce₂Fe₁₇ composition (89.5 at.% Fe) reported by Zarechnyuk & Kripyakevich (1963). The powder diffraction patterns of the RFe₇ phases can be indexed successfully by assuming these phases to be isostructural to Th₂Zn₁₇. This appears to be the basis for the assignment of the formulas Pr₂Fe₁₇ (Kripyakevich & Frankevich, 1966) and Nd₂Fe₁₇ (Savitskii *et al.*, 1965) since no supporting analytical data are included in either paper.

Kripyakevich, Terekhova, Zarechynuk & Burov (1963) report a Gd-Fe intermediate phase of the Th₂ Zn₁₇ type to be the main component of a Gd-Fe alloy containing 86.5 at.% Fe, and a Gd-Fe intermediate phase of the Th₂Ni₁₇ type to be the main component of an alloy containing 89.5 at.% Fe. While the authors do not indicate the nature of the deviation from A₂B₁₇ stoichiometry in the Gd-Fe phase having the Th₂Zn₁₇ type structure, it appears possible that this phase may be isostructural with the RFe₇ phases reported here.

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