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A re-examination of the Kraft and Flinn diffraction data for Cr_2B, $(Cr,Fe)_2B$, and the boride phase in Fe + 18.5 wt % Ni + 20 wt % Cr + B alloys

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Kraft and Flinn's [2] powder X-ray diffraction data obtained from excess-phase particles in Fe + 18.5 wt% Ni + 20 wt% Cr + B alloys have been analyzed. The major boride phase was determined to be $(Cr,Fe)_2B$ (orthorhombic, Fddd, isomorphous with Cr_2B). Its lattice parameters, $a = 1.4585 \pm 0.0010$ nm, $b = 0.7331 \pm 0.0005$ nm, $c = 0.4223 \pm 0.0003$ nm, are indicative of the composition $(Cr_{1.04\pm0.08}Fe_{0.96\pm0.08})B$. It was not possible to identify the phase(s) giving rise to a much weaker set of diffraction lines in the patterns. Of the two alloys used by Kraft and Flinn as standards, the one intended to be Cr_2B was found to be a mixture of Cr_2B and Cr_5B_3. The second, intended to be $Cr_{1.0}Fe_{1.0}B$ was determined to be $Cr_{0.67\pm0.07}Fe_{1.33\pm0.07}B$. © *1999 Kluwer Academic Publishers*

1. Introduction

During a recently completed investigation of boride phases in cast nickel-base alloys [1], a study by Kraft and Flinn (hereafter denoted K/F) concerning boride phases in austenitic alloys [2] was reexamined. In that study, K/F extracted excess phase particles from as-cast Fe+18.5%Ni+20%Cr alloys that contained 0.32, 0.80, and 1.49 wt% B. They obtained chromium-radiation powder diffraction patterns from the excess phase particles extracted from these alloys and from pulverized quantities of two specially prepared alloys (denoted S1 and S2). S1 had the intended composition Cr_2B while S2, was intended to be $Fe_{1.0}Cr_{1.0}B_{1.0}$.

Although Aronsson and Aselius [3] had determined the structure (orthorhombic) and lattice parameters of Cr_2B in 1958 from single crystal data, K/F were apparently unaware of this result. Since there was no powder pattern for Cr_2B available, they assumed the one obtained by themselves from S1 to be correct. When this pattern was compared to ones obtained from S2 and from the extracts, many common lines were observed. On this basis K/F concluded that Cr_2B was the major boride phase present in S2 and in the Fe+Ni+Cr+B alloys as well. A problem arose when a number of additional, and generally much weaker, lines were found in the diffraction patterns obtained from the extracted borides. In order to account for these, K/F proposed the existence of two additional phases in the Fe+Ni+Cr+B alloys: CrB and Fe_2B.

A major shortcoming of the K/F work was the lack of indexing of any of the diffraction patterns. Doing so would have provided an accurate determination of which lines belonged to the major boride phase and which remained to be identified. It also would have made lattice parameter determinations possible. This could have led them to the realization that the major boride phase in S2, and that of the extracts, contained iron and chromium atoms. Other investigators [3–5] have shown that about two-thirds of the chromium atoms in Cr_2B can be replaced by iron. It is conventional to refer to such a phase as $(Cr,Fe)_2B$. It is known that the presence of even trace concentrations of boron in Fe+Ni+Cr austenites results in the precipitation of $(Cr,Fe)_2B$ particles. [6–8] Nickel, in contrast to iron, is known to be sparingly soluble in Cr_2B [4, 9].

The purpose of the present work was to re-examine the K/F diffraction data with the aim of (a) solving the patterns for S1 and S2, (b) establishing the fact that the dominant boride phase in the extracts from as-cast samples of the Fe+Ni+Cr+B alloys was $(Cr, Fe)_2B$, (c) determining the lattice parameters of this phase and by comparison with published lattice parameters attempt to establish its composition, then (d) obtaining an accurate list of the lines remaining to be identified in the patterns of the extracts. This list was used in attempts to determine whether any additional phases could be identified in the Fe+Ni+Cr+B alloy extracts.

2. Procedure: programmed solutions for diffraction data

The K/F diffraction data consisted of lists of *d*-spacings (in nm) for the various samples. As a first step, lines in the patterns from S1 and S2 believed to be due to the presence of Cr_2B or $(Cr,Fe)_2B$ were tentatively indexed. Then, the (*hkl*) and associated *d*-spacings were input to a weighted least-squares FORTRAN program [10] for calculating lattice parameters. The program, which is based largely on the algorithims of Hess [11], uses the Taylor–Sinclair/Nelson–Riley expression for the systematic errors function. Lines in the least-squares analysis were weighted on the basis of the intensities reported by K/F. For example, lines with VVS (very very strong), VS (very strong), and S (strong) intensities were weighted 1.0, those with m (medium) intensities were weighted 0.6 w (weak), 0.3 VW (very weak) 0.2, and VVW (very very weak) 0.1 or 0.0. The program was operated in the Cohen-analysis mode [11]. A wavelength of 0.2291 nm was used in all calculations. After solutions for the patterns reported for S1 and S2 had been completed, the data for the boride phase extracts were processed in the same manner as just described.

3. Results and discussion

3.1. Sample S1 (Intended to be Cr_2B)

The solution for the powder pattern data from sample S1 is given in Table I. The sample was found to be a mixture of two phases: Cr_2B (orthorhombic, space group Fddd) and Cr_5B_3 (tetragonal, space group 14/mcm). On the basis of the line intensities reported by K/F, the amounts of these phases is estimated to be in the approximate ratio 9 to 1. The presence of some Cr_5B_3 is not surprising since Cr_2B results from a peritectic reaction between a liquid phase containing 31 at % B and Cr_5B_3 at temperatures below about 1870 °C [12]. The lattice parameters (nm) were determined to be: (a) for the Cr_2B: $a = 1.4708 \pm 0.0004$, $b = 0.7419 \pm 0.0003$, and $c = 0.42500 \pm 0.00005$, and (b) for the C_5B_3: $a = 0.5452 \pm 0.0001$ and $c = 1.0089 \pm 0.003$. Given the limits of accuracy imposed by the 4 and 5 significant figure *d*-spacings reported by K/F (and in the case of Cr_5B_3, by its small volume fraction), these values are in good agreement with the most recently published data [13–16]. It is interesting to note that the unindexed K/F pattern for S1 served as a standard pattern for Cr_2B in the JCPD files until the mid-1980s [17].

The *d*(obs) values listed in Table I (nm) are those reported by K/F while the *d*(calc) values are based on the lattice parameters determined from the data set. All of the K/F patterns were obtained using an 11.46 cm diameter powder camera. The systematic error for this method, as is well known, has the effect of displacing the lower-angle lines to values greater than the actual diffraction angle. Correspondingly, the *d*-spacings for the lower angle lines are less than the true values. Only for (*hkl*) in the far-back-reflection region do the *d*(obs) and *d*(calc) values converge. This effect is apparent in Table I and will be in later tables as well.

The Cr_2B sample was prepared for K/F by R. Steinitz who prepared many such samples for other investigators (including one of the present authors) in the 1950s and 1960s. Its cleanliness is apparent from the fact that all lines reported by K/F could be accounted for as being due to either Cr_2B or Cr_5B_3. The average random error of measurement was smaller for this pattern than for any of the others examined here. Even so, individual (*hkl*) in a number of pairs and triplets (for example the (0 4 0)/(0 2 2), the (4 4 0)/(4 2 2), and the (8 4 0)/(8 2 2)/(9 3 1)), having sufficient angular separation to be read as separate lines, were not resolved. In instances where this occurred, the lines were usually zero-weighted in the least-squares analysis.

TABLE I Indexed Cr-radiation powder pattern of sample S1[b]

		Cr_2B		Cr_5B_3	
d(obs) (nm)	*I*(obs)	(*hkl*)	*d*(calc) (nm)	(*hkl*)	*d*(calc) (nm)
0.365	w	4 0 0	0.3675		
0.357	w	1 1 1	0.3577		
0.303	vvw			1 1 2	0.3063
0.293	m	3 1 1	0.2946		
0.271	vvw			2 0 0	0.2726
0.2504	vw			0 0 4	0.2522
0.2381	vvw			2 0 2	0.2398
0.2356	vw			2 1 1	0.2370
0.2286	vs	5 1 1	0.2299		
0.2107	vvs	1 3 1	0.2115	1 1 4	0.2111
0.2038	vvs	2 0 2	0.2044		
0.2038	—	6 2 0	0.2041		
0.1965	w			2 1 3	0.1974
0.1950	m	3 3 1	0.1959		
0.1841	m	0 4 0	1.854	2 0 4	0.1851
0.1841	—	0 2 2	0.1843		
[a]		8 0 0	0.1838		
0.1821	s	7 1 1	0.1825		
0.1720	w			3 1 0	0.1724
0.1678	vw			0 0 6	0.1681
0.1649	vs	4 4 0	0.1656		
0.1649	—	4 2 2	0.1648		
0.1489	w	9 1 1	0.1494		
0.1391	vvw	1 5 1	0.1394		
0.1344	vw	3 5 1	0.1347		
0.1336	vw	3 1 3	0.1338		
0.1300	—	8 4 0	0.1305		
0.1300	—	8 2 2	0.1302		
0.1300	—	9 3 1	0.1298		
0.1263	s	5 5 1	0.1264		
0.1256	s	5 1 3	0.1258		
0.1256	—	11 1 1	0.1257		
0.1240	w			2 1 7	0.1241
0.1230	w			4 1 3	0.1231
0.1224	vvs	12 0 0	0.1225		
0.1224	—	1 3 3	0.1225		
0.1219	s	2 6 0	0.1219		
0.1214	vs	6 4 2	0.1214		
0.1209	s	10 0 2	0.1209		
0.1192	m	3 3 3	0.1192		
0.1165	vs	7 5 1	0.1165		
0.1160	vs	7 1 3	0.1160		

[a] Not reported.

[b] Because of interference, the following lines were zero-weighted in the analysis of the data for Cr_2B the (1 3 1), (2 0 2)/(6 2 0), (0 4 0)/(0 2 2), (4 4 0)/(4 4 2), and the (8 4 0)/(8 2 2)/(9 3 1); for Cr_5B_3 the (1 1 4) and the (2 0 4).

3.2. Sample S2 (Intended to be $Cr_{1.0}Fe_{1.0}B_{1.0}$)

As shown in Table II, it was possible to index 18 lines in the pattern for sample S2 on an orthorhombic basis

TABLE II Indexing and diffracted intensities for Cr-radiation powder pattern of sample S2 (intended to be $Cr_1Fe_1B_1$)[d]

(*hkl*)	*I*(obs)	*d*(obs) (nm)	*d*(calc) (nm)	*I* for Mn_2B
4 0 0	w	0.363	0.3635	5.3
1 1 1	w	0.353	0.3540	5.2
3 1 1	w	0.291	0.2916	4.9
5 1 1	s	0.227	0.2275	28.9
1 3 1	vs	0.2083	0.2086	45.6
2 0 2/6 2 0	vvs	0.2020	0.2204, 0.2022	30.8, 30.5
3 3 1	m	0.1933	0.1933	14.5
0 4 0/0 2 2	w	0.1823	0.1826, 0.18255	2.3, 3.5
8 0 0	—	[a]	0.1820	1.5
7 1 1	m	0.1802	0.1806	20.1
5 3 1	—	[a]	0.1710	0.5
4 4 0/4 2 2	s	0.1633	0.1632, 0.1631	7.5, 17.6
9 1 1	vw	0.1477	0.1478	2.7
1 5 1/1 1 3	vvw	0.1374	0.1374, 0.1373	0.2, 0.5
3 5 1/3 1 3	vvw	0.1329	0.1328, 0.1328	1.1, 0.7
8 4 0/8 2 2	m	0.1288	0.1288, 0.1288	4.7, 11.5
9 3 1	—	[a]	0.1282	4.0
5 5 1/5 1 3	s	0.1246	0.1247, 0.1246	12.1, 10.5
11 1 1	—	[a]	0.1243	1.3
12 0 0/1 3 3	vs	0.1213	0.1213, 0.1212	14.4, 26.5
2 6 0/6 4 2	vvs[b]	0.1200	0.1201, 0.1199	21.5, 39.1
10 0 2	—	[a]	0.1197	18.9
3 3 3	m	0.1180	0.1180	15.2
7 5 1	—	[c]	0.1150	100.0
7 1 3	—	[c]	0.1149	93.5

[a] Not reported.

[b] Broad lines; may also include the (10 0 2).

[c] Diffraction angle is greater than about 85°. Line was either too close to the incident-beam collimator to read or actually impinged on the collimator.

[d] Unindexed lines with the following *d*-spacings are not included: 0.276 (vw), 0.234 (w), 0.1958 (w), 0.1427 (vw), 0.1255 (w), and 0.1163 (m).

(space group Fddd), that is, as a phase isomorphous with Cr_2B. The lattice parameters (in nm) were determined to be $a = 1.4538 \pm 0.0005$, $b = 0.7304 \pm 0.0001$, and $c = 0.42140 \pm 0.00005$. The last two columns in Table II compare the visually estimated line intensities reported by K/F with those calculated here for a powder pattern of Mn_2B [18]. The latter has the same structure and nearly the same lattice parameters, positional co-ordinates, and scattering factors as $(Cr,Fe)_2B$ alloys. In calculating the Mn_2B diffracted intensities, the scattering factors for manganese and boron atoms were corrected for dispersion and the Debye–Waller factor was assumed to be 1.0. The agreement between the two sets of intensities is satisfactory.

3.3. $(Cr,Fe)_2B$ in the Fe+Ni+Cr+B alloys

It was possible to index 15, 19, and 20 lines, respectively, in the boride phase patterns obtained from the 0.32 wt % B, 0.80 wt % B, and 1.49 wt % B samples as due to the presence of the $(Cr,Fe)_2B$ phase. The lattice parameters (nm) in each case were found to be as follows: $a = 1.4585 \pm 0.0010$, $b = 0.7331 \pm 0.0005$, and $c = 0.4223 \pm 0.0003$. At this point in the present investigation, it was clear that the lattice parameters of the Cr_2B phase in alloy S1 were greater than those of the $(Cr,Fe)_2B$ phase in the Fe+Ni+Cr+B alloys. The latter were, in turn, greater than those of the $(Cr,Fe)_2B$ in alloy S2.

3.4. Estimating the compositions of the $(Cr,Fe)_2B$ phases

The next task was to attempt to estimate the compositions of the $(Cr,Fe)_2B$ in S2 and in the extracts. Subsequent to the K/F work, other investigators found that the *a*, *b*, and *c* values of the Cr_2B unit cell decrease when chromium atoms are replaced by iron [3–5]. Were the functional dependence of *a*, *b*, and *c* on the iron atom concentration in this phase well established, the present lattice parameter values could be used to estimate the composition of the $(Cr,Fe)_2B$ in S2 and in the Fe+Ni+Cr+B alloy extracts (provided the latter's values are not measurably affected by dissolved nickel atoms). Unfortunately, this relationship has not been established. In lieu of this, curves were constructed assuming a linear relation for *a*, *b*, and *c* on the iron concentration in this phase. The data used for this purpose were the *a*, *b*, and *c* values for Cr_2B, as noted in Section 3.1, and the Brown and Beernsten result obtained from a single crystal of $Cr_{0.9}Fe_{1.1}B_{0.9}$ [4]. They found the lattice parameters (nm) of their crystal be $a = 1.457$, $b = 0.732$, and $c = 0.422$. These values are in relatively good agreement with $a = 1.458$, $b = 0.733$, and $c = 0.421$ reported by Aronsson and Aselius for a boride with nearly the same Cr:Fe atom ratio [3].

The lines marked a, b, and c in Fig. 1 have been drawn to pass through points in the two data sets noted above. The intercepts of the lattice constants of sample S2 on these lines occur within the right-most shaded region. Thus, the composition of the $(Cr,Fe)_2B$ phase in S2 is indicated to be $Cr_{0.67 \pm 0.07}\,Fe_{1.33 \pm 0.07}B$. The intercepts of the lattice constants of the $(Cr,Fe)_2B$ phase extracted from the Fe+Ni+Cr+B alloys occur within the composition range defined by the left-most shaded region. Thus, these borides are indicated to be $Cr_{1.04 \pm 0.08}Fe_{0.96 \pm 0.08}B$.

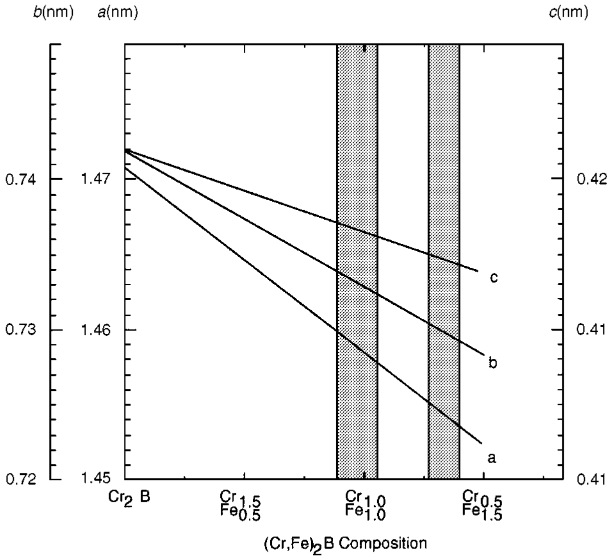

Figure 1 The lines a, b, and c mark the dependence (assumed linear) of the lattice parameters of the $(Cr,Fe)_2B$ phase on concentration. The right-most shaded region contains the lattice parameter values for boride-phase particles extracted from the Fe+Ni+Cr+B alloys and gives an estimated mean composition of $Cr_{0.66}Fe_{1.34}B$. The left-most shaded region contains the lattice parameter values for the alloys S1 and indicates a mean composition of $Cr_{1.04}Fe_{0.96}B$.

3.5. Unidentified lines in patterns from the extracts

A number of lines in the patterns of the extracts remained unindexed after accounting for the $(Cr,Fe)_2B$ phase. These numbered 18 (out of a total of 33 lines) for the extract from the 0.32% B alloy, 9 (out of 28 lines) for the 0.80% B alloy, and 5 (out of 25 lines) for the 1.49% B alloy. Only one of these lines was listed as vs, one as s, and four as m in intensity. The remaining 26 were w, vw, or vvw. Micrographs obtained by K/F revealed a small concentration of a dark-etching phase they believed responsible for these lines [2].

None of the unidentified lines in these patterns could be attributed to the presence of either Cr_5B_3 or undissolved volumes of the matrix phase (austenite). Only four lines (at $d = 0.333$ nm, $d = 0.160$, 0.137, and 0.115 nm) were common to patterns from the three as-cast alloy extracts. Although K/F suggested that the unindexed lines could be ascribed to the presence of two additional phases, CrB (orthorhombic, space group Cmcm) and Fe_2B (tetragonal, space group 14/mcm), their failure to index any of the patterns prevented an accurate compilation of unindexed lines.

Because the extract pattern from the borides obtained from the 0.32 wt % B alloy contained the greatest number of unindexed lines, it was selected for further analysis. All of the *d*-spacings in this pattern are listed in Table III. The (*hkl*) assigned to lines attributable to $(Cr,Fe)_2B$ are given in column 4. The entire list of *d*-spacings, including those of unindexed and already indexed lines, was then compared with *d*-spacings and intensities in powder patterns. These were (a) calculated by us using published single crystal data [19, 20], and (b) available in the literature for phases of the following types: Cr_xB_y, $(Cr,M)_xB_y$, Fe_xB_y, $(Fe,M)_xB_y$, Ni_xB_y, $(Cr,Ni)_xB_y$, $(Fe,Ni)_xB_y$, Cr_xC_y, and Fe_xB_y where M denoted Cr, and/or Fe, and/or Ni [20, 21]. It was necessary to include carbides and borocarbides because the K/F alloys contained 0.03 to 0.05 wt % C. Confirmation was not obtained for the presence of any of these phases. The diffraction data do not support the presence of either CrB or Fe_2B in these

TABLE III Cr-radiation pattern of extract from 0.32% B alloy [a]

			$(Cr,Fe)_2B$		Cubic		Hexagonal		Orthorhombic	
	d(obs) (nm)	*I*(obs)	(*hkl*)	*d*(calc) (nm)	(*hkl*)	*d*(calc) (nm)	(*hkl*)	*d*(calc) (nm)	(*hkl*)	*d*(calc) (nm)
1	0.425	w					2 0 1	0.4251	2 1 0	0.423
2	0.404	w							3 0 0	0.405
3	0.3618	vw	4 0 0	0.3651						
4	0.3333	vs			2 2 2	0.3337	2 2 0	0.3338	3 1 0	0.3336
5	0.2947	vvw					2 2 1	0.2947	0 2 0	0.2943
6	0.2805	w			4 1 0	0.2803			2 0 1	0.2790
7	0.2652	w			3 3 1	0.2652	3 2 0	0.2652	2 2 0	0.2649
8	0.2515	s					4 1 0	0.2523	2 1 1	0.2521
9	0.2464	w			3 3 2	0.2464				
10	0.2270	vs	5 1 1	0.2283						
11	0.2169	vvw					5 0 1	0.2170		
12	0.2089	s	1 3 1	0.2092			0 0 3	0.2091		
13	0.2025	s	2 0 2	0.2029			3 2 2	0.2025	2 2 1	0.2025
	0.2025	—	6 2 0	0.2027					6 0 0	0.2025
14	0.1949	w								
15	0.1810	m	7 1 1	0.1813						
16	0.1668	w			4 4 4	0.1668	4 4 0	0.1669	6 2 0	0.1668
17	0.1635	m	4 4 0	0.1637					6 1 1	0.1635
	0.1635	—	4 2 2	0.1637						
18	0.1604	w			6 4 0	0.1603	6 2 0	0.1603	2 3 1	0.1605
19	0.1540	vw					6 1 2	0.1537	3 3 1	0.1539
20	0.1480	w	9 1 1	0.1484	6 4 3	0.1480	5 4 0	0.1480		
21	0.1461	vvw					5 3 2	0.1461	1 4 0	0.1461
22	0.1428	vw					6 2 2	0.1428	2 4 0	0.1430
23	0.1385	m					5 2 3	0.1386	0 2 2	0.1386
24	0.1372	m							5 3 1	0.1373
25	0.1292	m	8 4 0	0.1293	8 4 0	0.1292				
	0.1292	—	8 2 2	0.1292						
26	0.1289	vvw	9 3 1	0.1288			7 2 2	0.1288		
27	0.1251	w	5 5 1	0.1251			5 1 4	0.1251		
28	0.1248	w	1 1 1 1	0.1249						
29	0.1218	s	1 2 0 0	0.1217	9 3 0	0.1218				
	0.1218	—	1 3 3	0.1215						
30	0.1206	vw					2 1 5	0.1206		
31	0.1204	w	2 6 0	0.1204					5 2 2	0.1204
	0.1204	—	6 4 2	0.1203						
32	0.1183	vs	3 3 3	0.1184						
33	0.1156	w			8 6 0	0.1156	1 0 0 0	0.1156	2 5 0	0.1156

[a] The *d*(calc) values were based on the following lattice parameters (nm): (a) for the cubic $a_0 = 1.1558$, (b) for the hexagonal $a = 1.3350$ and $c = 0.6273$, and (c) for the orthorhombic $a = 1.2145$, $b = 0.5886$, and $c = 0.3341$.

samples. While six lines associated with the $(Cr,Fe)_2B$ phase are close to lines expected for Fe_2B (lines 3, 15, 17, 24, 26, 31), none of the remaining lines expected for Fe_2B, including the three strongest lines for a Cr-radiation pattern, were reported by K/F.

Finally, attempts were made to find structures and lattice constants that could account for the maximum number of unidentified lines. The results are of sufficient interest to report here.

(a) The *d*-spacings of lines 4, 6, 7, 9, 16, 18, 20, 25, 29 and 33 listed in Table III, when multiplied by $(12)^{1/2}$, $(17)^{1/2}$, $(19)^{1/2}$, $(22)^{1/2}$, $(48)^{1/2}$, $(52)^{1/2}$, $(61)^{1/2}$, $(80)^{1/2}$, $(90)^{1/2}$, and $(100)^{1/2}$, respectively, yielded values in the 1.155 to 1.156 nm range. When these lines were input to the weighted least-squares program for a simple cubic structure the resulting lattice parameter was found to be 1.1558 nm. This does not correspond to any known phase in Fe+Ni+Cr+B+C alloy system.

(b) Columns 7 and 8 in Table III list the indices and d(calc) values for the best fit assuming a hexagonal structure. The resulting lattice parameters (nm) are $a = 1.3350$ and $c = 0.6273$. A total of 19 lines are accounted for including 14 of the unindexed group. This cell also fails to correspond to any known phase based on the constituent atoms.

(c) The last two columns in Table III assume the presence of a phase with an orthorhombic structure. The fit is quite good. All unindexed lines in the pattern save three (at $d = 0.2464$ (w), $d = 0.2169$ (vvw), $d = 0.1949$ (w), and $d = 0.1206$ (vw)) being accounted for. The indicated a, b, and c values were, respectively, 1.215, 0.589, and 0.314 nm. This cell also fails to correspond to any known phase comprised of these elements.

The above suggests that the phase(s) giving rise to the unindexed lines in the patterns of the extracts are either due to an impurity phase or to an impurity-stabilized phase. Evidence for the transient nature of this phase became apparent when the diffraction pattern reported by K/F for excess phase particles extracted from the 0.80% B sample after a 24 h homogenization at 1204 °C was analyzed. Of the twenty-one *d*-spacings in this pattern, seventeen indexed as $(Cr,Fe)_2B$ with lattice parameters (nm) as follows: $a = 1.4598$, $b = 0.7333$, $c = 0.42246$. These are essentially unchanged from values obtained from this phase in the as-cast sample. The remaining lines had *d*-spacings of 0.3528, 0.2512, 0.1207, and 0.1154 nm. Only one of the nine unindexed lines in the pattern for the as-cast boride sample (at $d = 0.1154$ nm) was also observed in the homogenized sample.

4. Conclusions

1. The major boride phase in the K/F Fe+18.5 wt % Ni+20 wt % Cr+B alloys was confirmed to be $(Cr,Fe)_2B$ with lattice parameters indicative of a composition given by $Cr_{1.04\pm0.08}Fe_{0.96\pm0.08}B$.

2. The unidentified lines in diffraction patterns obtained from the Fe+Ni+Cr+B alloy extracts do not derive, as suggested by K/F, from the presence of CrB and Fe_2B in these alloys. Although it was possible to obtain several good fits based on assumed structures (eg, hexagonal and orthorhombic) the resulting lattice parameters did not match those of known phases in this alloy system. These lines may derive from the presence of a small volume fraction of an impurity-stabilized phase.

3. The alloy specially prepared for the K/F investigation with the intended composition Cr_2B was found to be a two-phase aggregate of Cr_2B and Cr_5B_3. The Cr_2B was estimated to comprise about 90% of the sample volume.

4. Lattice parameter determinations made on a second alloy specially prepared for the K/F investigation indicate the alloy composition to have been $Cr_{0.66\pm0.07}Fe_{1.34\pm0.07}B$.

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