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New borides $Er_{0.917}Ni_{4.09}B$ and $ErNi_7B_3$ and their crystal structures

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Abstract

New borides have been synthesized and their crystal structures have been determined using X-ray single-crystal methods, namely: $Er_{0.917}Ni_{4.09}B$, own structure type, space group $P6/mmm$, $a = 14.8399(3)$, $c = 6.9194(3)$ Å, $R_F = 0.0545$, and $ErNi_7B_3$, own structure type, space group $I4_1/amd$, $a = 7.6577(2)$, $c = 15.5798(5)$ Å, $R_F = 0.0451$. The relationship between these structures and the structure types of $CeCo_4B$, $Y_{0.915}Ni_{4.12}B$ and $Sc_4Ni_{29}B_{10}$ has been discussed.

Keywords: Crystal structure; Erbium; Boride

1. Introduction

The Er–Ni–B phase diagram has been constructed by Cherniak [1], but after new borides have been revealed $Er_2Ni_{10}B_5$ [2], $Er_3Ni_{19}B_{10}$ [3], $Er_2Ni_{15}B_9$ [4], $ErNi_2B_2$ [5], and $Er_2Ni_3B_6$ [6] the diagram requires refinement and correction.

$ErNi_4B$ with the $CeCo_4B$-type structure was first reported in Ref. [7], but lattice parameters were not presented. Existence of this compound and its crystal structure were confirmed in Ref. [8] (space group $P6/mmm$, $a = 4.949(4)$, $c = 6.931(11)$ Å), but atomic coordinates were not refined. In the related Y–Ni–B system two borides of the approximate composition YNi_4B were found. Using X-ray powder method the crystal structure of the compound with the exact composition YNi_4B was found to be isotypic with the $CeCo_4B$-type structure, $a = 4.977$, $c = 6.942$ Å [7] or $a = 4.969(3)$, $c = 6.953(5)$ Å [9]. For the second boride parameters of the hexagonal unit cell were determined using single-crystal data ($a = 14.89$, $c = 6.91$ Å), but atomic coordinates were not determined [10]. The crystal structure of this compound was completely revealed in Ref. [11] and its exact composition was described by the formula $Y_{0.915}Ni_{4.12}B$ (space group $P6/mmm$, $a = 14.9085(10)$ $c = 6.9196(8)$ Å). Taking into account the similarity between the Y–Ni–B and Er–Ni–B systems, we decided to perform a single-crystal investigation of the $ErNi_4B$ structure.

Besides, in the Er–Ni–B system, a compound was obtained of approximate composition $Er_4Ni_{29}B_{10}$ closely related to the tetragonal structure $Sc_4Ni_{29}B_{10}$ (space group $I4_1/amd$, $a = 7.665(1)$, $c = 15.584(3)$ Å) [12]. This structure was determined by X-ray powder method, and also needed confirmation using single-crystal data.

So, the single-crystal investigation of the crystal structure of the borides $\sim ErNi_4B$ and $\sim Er_4Ni_{29}B_{10}$ was the goal of the present work.

2. Experimental details and structure determination

Samples with the nominal compositions $ErNi_4B$ and $Er_4Ni_{29}B_{10}$ and a total mass of about 2 g each were prepared from mixtures of compact erbium (99.9 wt%) and powders of nickel (99.98 wt%) and boron (99.4 wt%). The powders of Ni and B were previously pressed into pellets and were then melted together with pieces of Er in an arc furnace with a tungsten electrode in a purified argon atmosphere. Gray single crystals with metallic luster of the $ErNi_4B$ boride (plates) and of the $Er_4Ni_{29}B_{10}$ boride (needles) were extracted from the alloys after grinding. Conditions of the single-crystal data collection are listed in Table 1.

Data collection strategy was performed with the help of the program COLLECT [13] and reflections were

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Table 1
Data collection and refinement for $Er_{0.917}Ni_{4.09}B$ and $ErNi_7B_3$

Empirical formula	$Er_{0.917}Ni_{4.09}B$	$ErNi_7B_3$
Space group	$P6/mmm$ (No. 191)	$I4_1/amd$ (No. 141)
Unit-cell dimensions (Å)	$a = 14.8399(3)$	$a = 7.6577(2)$
	$c = 6.9194(3)$	$c = 15.5798(5)$
Z	18	8
Cell volume (Å^3)	1319.66(7)	913.61(8)
Calculated density (g/cm^3)	9.162(1)	8.879(1)
Diffractometer	Kappa CCD-Nonius	
Radiation and wavelength	Mo$K\alpha$, 0.71073	
Number of atoms in cell	108.2	88
Number of atom sites	20	7
Number of free parameters	68	36
Mode of refinement	$F(hkl)$	
Restrictions	$\lvert F_{h\,k\,l}\rvert > 4.00\sigma(F)$	
2ϑ and $\sin\vartheta/\lambda$(max)	54.91°, 0.649	74.04°, 0.847
Index ranges	$-19 \leqslant h \leqslant 19$, $-19 \leqslant k \leqslant 17$, $-8 \leqslant l \leqslant 8$	$-12 \leqslant h \leqslant 9$, $-12 \leqslant k \leqslant 11$, $-26 \leqslant l \leqslant 26$
Reflections collected	4915	1436
Number of non equivalent reflections	592	604
Extinction formalism	Sheldrick-1, 0.000085	
Final R indices		
R_F	0.0545	0.0451
R_w	0.0537	0.0446
R_{int}	0.10	0.089
R_σ	0.039	0.032
Goodness-of-fit on F	1.040	1.020
Largest diff. peak/hole	4.3/–3.0 e/Å^3	6.9/–2.4 e/Å^3

$R_\sigma = \sum\sigma F_o^2/\sum F_o^2$.
$R_F = \sum||F_o| - |F_o||/\sum F_o$.
$R_w = 1/\sum F_c^2 + 0.004F_o^2$
GoF $= \left(\sum w(F_o^2 - F_c^2)^2/(n-p)\right)^{1/2}$ with $w = 1/(\sigma^2(F_o^2) + (a \times P)^2 + b \times P)$, n is a number of observed reflections, p is a number of parameters refined

Note. $T = 293(2)$ K, graphite monochromator, the refinement method was full-matrix least-squares on F.

corrected using the program DENZO of the Kappa CCD software package [14]. Owing to the small size of the single crystals, no absorption correction was necessary. The presence of a center of symmetry and space group was checked using the procedure E-STATS [15] and ABSEN [16] of the WinGX-V1.64.04 [17] software. The crystal structure was solved in space group $P6/mmm$ by direct methods (SIR 97 [18]) and least-squares refinements, difference Fourier syntheses were run with SHELXL-97 [19] and WinCSD [20] software.

3. Results and discussion

3.1. The $Er_{0.917}Ni_{4.09}B$ structure

The crystal structure was solved by direct methods; atomic coordinates and their thermal parameters are listed in Tables 2 and 3. The peculiarity of this structure is the presence of a number of crystallographic sites, which are partially occupied, partially those positions that are situated along the directions $0\,0\,z$ (Er4, Ni8-Ni12) and also $1/3\,2/3\,z$ (Er3). The Er4 atoms occupy the position 1(a) 0 0 0 only for 61.5%, and Ni atoms (Table 2) occupy the vacancies on the [001] axis.

A close relationship is observed between the structures of $Er_{0.917}Ni_{4.09}B$ and $Y_{0.915}Ni_{4.12}B$ [11]. In former compound the Ni atoms, partially occupy two additional positions (Ni8 and Ni12) (Table 2) compared to yttrium structure. The total number of atoms with $0\,0\,z$ coordinates is approximately the same, that is 2.68 atoms in $Y_{0.915}Ni_{4.12}B$ (0.465 Y and 2.21 Ni) and 2.78 atoms in $Er_{0.917}Ni_{4.09}B$ (0.615 Er and 2.16 Ni). Supposing, that the lattice parameter c is defined by the number and sizes of atoms in [001] direction, then c should be equal to $\sim$7.61 Å for the $Y_{0.915}Ni_{4.12}B$ structure and $\sim$7.51 Å for the $Er_{0.917}Ni_{4.09}B$ structure ($r_Y = 1.776$ Å, $r_{Er} = 1.734$ Å, $r_{Ni} = 1.246$ Å [21]). In reality, c is almost the same for both structures (6.92 Å). It may indicate stronger compression between atoms in [001] direction in the erbium containing structure.

All boron atoms in the $Er_{0.917}Ni_{4.09}B$ structure are isolated from each other and are situated in the centers of trigonal prisms formed by metal atoms. These trigonal prisms are connected by edges and form hexagonal rings (Fig. 1). Coordination number (CN) of boron atoms is 6+3 (six metal atoms form the trigonal prism and three atoms center the rectangular

Table 2
Coordinates and thermal parameters of atoms, occupancy factors (G), and atomic arrangement for $Er_{0.917}Ni_{4.09}B$ structure (space group $P6/mmm$)

Atom	Position	G	x	y	z	B_{iso} (Å^2)	Atomic arrangement[a]
Er1	6(k)	1	0.32170(6)	0	1/2	0.72(2)	2Er 14Ni 4B
Er2	6(j)	1	0.34390(6)	0	0	0.74(2)	2Er 16Ni 2B
Er3	2(c)	0.949(5)	1/3	2/3	0	0.77(4)	2Er 18Ni
Er4	1(a)	0.615(9)	0	0	0	0.76(8)	12Ni
Er5	2(d)	1	1/3	2/3	1/2	0.66(3)	2Er 12Ni 6B
Ni1	24(r)	1	0.16562(8)	0.49803(8)	0.2976(2)	0.68(4)	4Er 7Ni 2B
Ni2	12(o)	1	0.16538(6)	2 ×	0.2667(3)	0.91(5)	4Er 6Ni 2B
Ni3	12(n)	1	0.1708(1)	0	0.1883(3)	0.97(5)	3Er 7Ni 1B
Ni4	6(m)	0.922(7)	0.0934(1)	2 ×	1/2	1.13(8)	2Er 8Ni
Ni5	6(l)	1	0.2238(1)	2 ×	0	1.07(7)	3Er 8Ni 1B
Ni6	6(l)	1	0.5603(1)	0.1206	0	1.04(7)	3Er 8Ni
Ni7	6(i)	1	1/2	0	0.2859(4)	0.74(6)	4Er 6Ni 2B
Ni8	2(e)	0.18(1)	0	0	−0.117(4)	0.9(4)	12Ni
Ni9	2(e)	0.38(1)	0	0	0.346(2)	1.1(2)	1Er 12Ni
Ni10	2(e)	0.16(1)	0	0	0.393(5)	0.8(4)	1Er 12Ni
Ni11	2(e)	0.265(9)	0	0	0.048(2)	0.8(3)	1Er 6Ni
Ni12	1(b)	0.19(1)	0	0	1/2	1.2(6)	2Er 6Ni
B1	6(m)	1	0.2113(9)	2 ×	1/2	1.4(4)	3Er 6Ni
B2	6(m)	1	0.5541(8)	0.1082	1/2	1.2(4)	3Er 6Ni
B3	6(l)	1	0.1239(8)	2 ×	0	0.8(4)	2Er 7Ni

[a] In coordination sphere only the atoms occupying more than 50% of sites were taken into account.

Table 3
Anisotropic thermal parameters (B, Å^2) for $Er_{0.917}Ni_{4.09}B$

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Er1	0.74(2)	0.54(3)	0.81(5)	$1/2B_{22}$	0	0
Er2	0.75(2)	0.70(3)	0.76(5)	$1/2B_{22}$	0	0
Er3	0.77(4)	B_{11}	0.76(8)	$1/2B_{11}$	0	0
Er4	0.48(7)	B_{11}	1.3(2)	$1/2B_{11}$	0	0
Er5	0.66(3)	B_{11}	0.76(8)	$1/2B_{11}$	0	0
Ni1	0.58(4)	0.62(4)	0.88(6)	0.33(4)	−0.06(4)	−0.01(4)
Ni2	0.65(5)	0.66(6)	1.43(10)	$1/2B_{22}$	−0.05(3)	$2B_{13}$
Ni3	0.88(5)	0.47(6)	1.43(11)	$1/2B_{22}$	−0.03(5)	0
Ni4	1.08(8)	1.38(11)	1.0(2)	$1/2B_{22}$	0	0
Ni5	1.04(7)	1.58(10)	0.77(14)	$1/2B_{22}$	0	0
Ni6	1.05(7)	1.67(10)	0.60(13)	$1/2B_{22}$	0	0
Ni7	0.59(6)	0.50(8)	1.11(12)	$1/2B_{22}$	0	0

$T = \exp(-1/4(B_{11}a^*2h2 + \cdots + 2B_{23}b^*c^*kl))$.

faces). Ni atoms in the completely occupied crystallographic sites have CN from 10 to 13. Ni atoms in partially occupied positions have the same CN, except for Ni11 and Ni12 atoms which have a smaller CN (7 and 8, respectively). It should be noted, that Ni atoms in partially occupied positions in any case have no boron atoms in their coordination polyhedra (CP). Similar to $CaCu_5$ and $CeCo_3B_2$ structure types, erbium atoms in the $Er_{0.917}Ni_{4.09}B$ structure have CN 20, and only CN of the Er3 atom decreases to 12. Er atoms in the partially occupied positions have no boron atoms in their coordination spheres. Moreover, the coordination sphere of Er3 does not include any atoms from partially occupied positions.

Interatomic distances (δ) in the $Er_{0.917}Ni_{4.09}B$ are listed in Tables 4 and 5. Shortening of the interatomic distances is insignificant, and is within 5% of the respective sum of the atomic radii. Only between Er3 and Ni3 atoms shortening of the distances increases to 8%. Such slight shortening of the interatomic distances is typical for boride structures [22].

Both structures $Er_{0.917}Ni_{4.09}B$ and $Y_{0.915}Ni_{4.12}B$ are members of the homologous series of the structures derived from the $CaCu_5$ and $CeCo_3B_2$ types (Fig. 2). Up to now five homologous series of structures derived from these two types are known. The planar nets that are perpendicular to the [00Z] direction form all these structures. Such nets are peculiar of the $CaCu_5$ and $CeCo_3B_2$ structures. The number of planar nets in the structure (Fig. 2) determines characteristic values of the lattice parameter c, while the value of the parameter a is nearly constant in different structures of this series.

Parameters a in the structures of the $Er_{0.917}Ni_{4.09}B$ and $Y_{0.915}Ni_{4.12}B$ are three times larger than those for the $CaCu_5$ and $CeCo_3B_2$ structures. Parameter c has a value as it was established for the $CeCo_4B$ structure: $c = c_{CaCu_5} + c_{CeCo_3B_2}$ (Fig. 2).

The absence of a RE atom in the position (001/2) and the small value of the occupancy factor for position (000) lead to the fact that the channel formed by Ni atoms along the [00Z] direction is filled only by atoms of partially occupied positions. Thus, ~1.4 of RE atoms are replaced by ~2.1 of Ni atoms. Similar substitution of RE atoms by the atoms of transition metals is observed in other hexagonal structures related to the $CaCu_5$-type, in particular, in the structures of $YNi_{9.41}$,

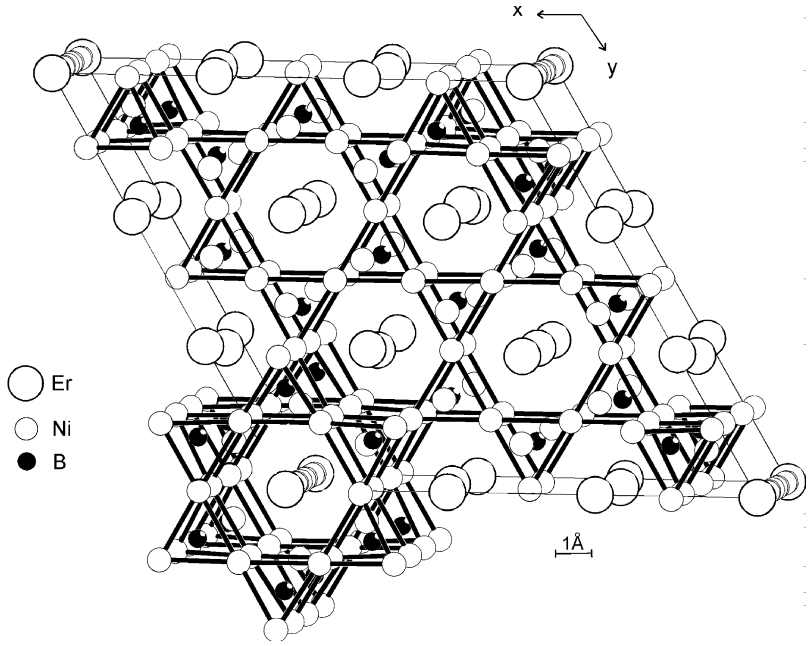

Fig. 1. Mode of connection of trigonal prisms centered by boron atoms in the $Er_{0.917}Ni_{4.09}B$ structure.

Table 4
Minimum interatomic distances (δ) in $Er_{0.917}Ni_{4.09}B$ structure for the atoms occupying more than 50% of the position

Atoms	δ (Å)	Atoms	δ (Å)
Er1–2 Er2	3.4754(3)	Ni3–1 Er4	2.850(2)
Er1–4 Ni2	2.884(1)	Ni3–2 Ni2	2.475(1)
Er1–2 B1	2.717(12)	Ni3–1 B3	2.070(8)
Er2–2 Er1	3.4754(3)	Ni4–2 Er1	2.950(2)
Er2–2 Ni6	2.786(2)	Ni4–2 Ni4	2.401(2)
Er2–2 B3	2.835(12)	Ni5–1 Er3	2.816(1)
Er3–2 Er5	3.4597(3)	Ni5–2 Ni2	2.379(2)
Er3–3 Ni6	2.735(2)	Ni5–1 B3	2.567(10)
Er4–12 Ni3	2.850(1)	Ni6–1 Er3	2.735(1)
Er5–2 Er3	3.4597(3)	Ni6–4 Ni1	2.482(2)
Er5–12 Ni1	2.861(1)	Ni7–2 Er1	3.032(1)
Er5–3B2	2.895(13)	Ni7–4 Ni1	2.474(1)
Ni1–1 Er5	2.862(2)	Ni7–2B2	2.031(8)
Ni1–1 Ni1	2.475(2)	B1–2 Er1	2.717(14)
Ni1–1 Ni7	2.474(1)	B1–2 Ni2	2.001(7)
Ni1–1 B2	2.023(10)	B2–1 Er5	2.895(11)
Ni2–2 Er1	2.884(2)	B2–4 Ni1	2.023(10)
Ni2–1 Ni5	2.379(2)	B3–2 Er2	2.835(13)
Ni2–1 B1	2.001(7)	B3–4 Ni3	2.070(10)

Table 5
Minimum interatomic distances (δ) between the atoms occupying less and more 50% of crystallographic position in $Er_{0.917}Ni_{4.09}B$ structure

Atoms	δ (Å)	Atoms	δ (Å)
Ni8–6 Ni3	2.582(6)	Ni10–1 Er4	2.620(3)
Ni9–1 Er4	2.397(14)	Ni10–6 Ni4	2.513(9)
Ni9–6 Ni4	2.626(6)	Ni10–6 Ni3	2.903(15)
Ni9–6 Ni3	2.760(6)	Ni11–6 Ni3	2.714(6)
		Ni12–2 Er4	3.4597(3)
		Ni12–6 Ni4	2.401(2)

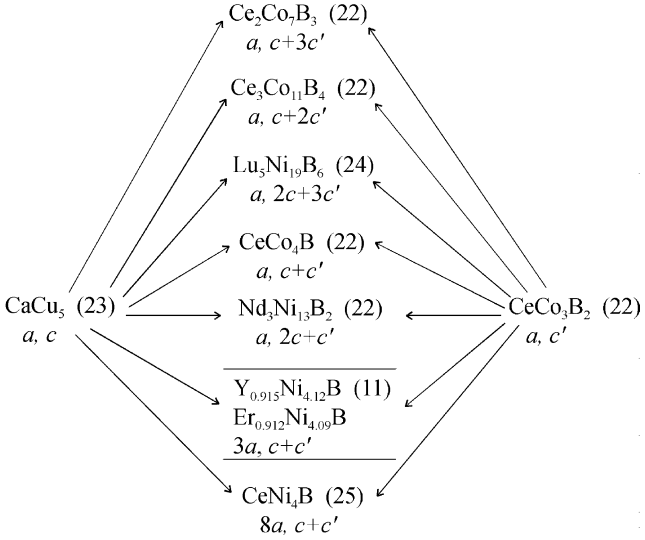

Fig. 2. Homologous series of the structure types derived from $CaCu_5$ and $CeCo_3B_2$ structures (references are given in the brackets).

$ErCo_{9.0}$ [24], and $Dy_{1.75}Ag_{8.1}Al_{9.2}$ [25]. All these compounds crystallize in the Th_2Ni_{17} type structure that is closely related with the $CaCu_5$-type [23].

3.2. The $ErNi_7B_3$ structure

The single-crystal investigation of the $\sim Er_4Ni_{29}B_{10}$ structure showed that symmetry and lattice parameters of this boride were in a good agreement with the data obtained by the X-ray powder method [12]. The crystal structure was solved by direct methods, atomic positional and thermal parameters are listed in Table 6, and

Table 6
Atomic, thermal parameters and atomic arrangement for $ErNi_7B_3$ boride (space group $I4_1/amd$)

Atom	Position	x	y	z	B_{iso} (Å^2)	B_{11}	B_{22}	B_{33}	Atomic arrangement[a]
Er	8(*e*)	0	0	0.28388(5)	0.37(1)	0.33(2)	0.39(3)	0.38(2)	14Ni 4B
Ni1	8(*c*)	0	1/4	1/8	0.38(4)	0.35(7)	0.43(7)	0.37(6)	2Er 8Ni 4B
Ni2	16(*g*)	0.1886(2)	×	0	0.50(2)	0.54(3)	B_{11}	0.43(4)	2Er 8Ni 4B
Ni3	16(*h*)	0	0.2874(2)	0.3932(1)	0.44(3)	0.40(5)	0.48(5)	0.45(5)	2Er 7Ni 3B
Ni4	16(*h*)	0	0.1611(2)	0.5560(1)	0.43(3)	0.49(5)	0.37(5)	0.41(5)	2Er 9Ni 2B
B1	8(*c*)	0	0	0.9422(2)	0.5(3)	0.1(6)	0.8(6)	0.7(6)	8Ni 1B
B2	16(*f*)	0.289(2)	1/4	1/8	0.4(2)	0.6(4)	0.2(3)	0.2(3)	2Er 7Ni

[a] $B_{12} = B_{13} = B_{23} = 0$.

Table 7
Interatomic distances (δ) for $ErNi_7B_3$ structure

Atoms	δ (Å)	Atoms	δ (Å)
Er–2 Ni4	2.618(2)	Ni4–2 B2	2.060(11)
Er–2 Ni3	2.730(2)	Ni4–1 Ni4	2.467(3)
Er–2 Ni3	2.783(2)	Ni4–2 Ni4	2.468(2)
Er–2 Ni4	2.783(2)	Ni4–1 Ni4	2.545(3)
Er–4 Ni2	2.837(2)	Ni4–1 Er	2.618(2)
Er–4 B2	2.882(8)	Ni4–2Ni3	2.644(2)
Er–2 Ni1	3.1292(8)	Ni4–1 Ni3	2.714(3)
		Ni4–1 Er	2.783(2)
Ni1–2 B1	2.182(10)	Ni4–2 Ni2	2.788(2)
Ni1–2 B2	2.209(14)		
Ni1–4 Ni2	2.4697(9)	B1–1B1	1.80(3)
Ni1–4Ni3	2.529(1)	B1–2 Ni3	2.102(13)
Ni1–2 Er	3.1292(8)	B1–2 Ni1	2.182(10)
		B1–4Ni2	2.232(9)
Ni2–2 B2	2.144(5)		
Ni2–2 B1	2.232(9)	B2–2Ni3	2.020(4)
Ni2–2 Ni1	2.4697(9)	B2–2Ni4	2.060(11)
Ni2–2 Ni3	2.664(2)	B2–2Ni2	2.144(5)
Ni2–2 Ni4	2.788(2)	B2–1Ni1	2.209(14)
Ni2–2 Er	2.837(1)	B2–2Er	2.882(8)
Ni2–2Ni2	2.888(2)		
Ni3–2 B2	2.020(4)		
Ni3–1 B1	2.102(13)		
Ni3–2 Ni1	2.529(1)		
Ni3–2 Ni4	2.644(2)		
Ni3–2 Ni2	2.664(2)		
Ni3–1 Ni4	2.714(3)		
Ni3–2 Er	2.783(2)		

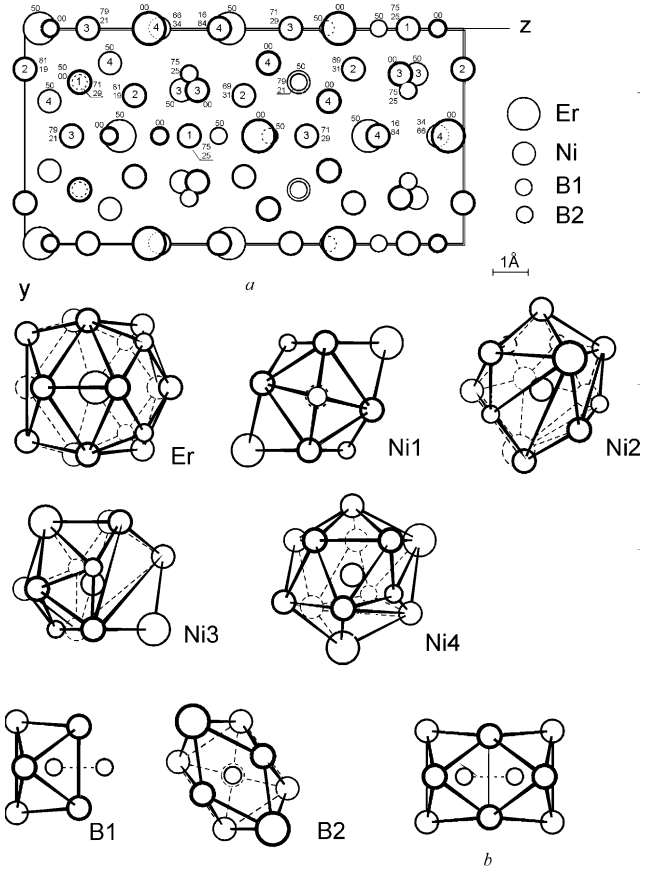

Fig. 3. The YZ projection of the $ErNi_7B_3$ structure (a) and CP of atoms; (b) coupled Archimedean cubes.

interatomic distances are given in Table 7. The compound has the composition described by the formula $ErNi_7B_3$. The YZ projection of the $ErNi_7B_3$ structure and CP of atoms are shown in Fig. 3. All boron atoms in this structure are at the centers of Archimedean cubes formed by the metal atoms (see Fig. 3). The Archimedean cubes centered by B1-atoms are connected with one another by square faces, forming B_2 pairs. B2-atoms are isolated one from another. Thus, according to the common tendency, the increasing boron content in the boride leads to the substitution of isolated boron atoms by B_2 pairs, B_n chains or boron frames [22]. In $Er_{0.917}Ni_{4.09}B$ (16.7 at% of B) boron atoms are isolated, but in the structure of $ErNi_7B_3$ (27.3 at% of B) the formation of B_2 pairs is observed. The main difference between the structures of $ErNi_7B_3$ and $Sc_4Ni_{29}B_{10}$ [12] is that in the former structure the coupled Archimedean cubes are centered solely by boron atoms, while in the latter one the coupled Archimedean cubes are centered by a pair B_2 (58% of Archimedean cubes) or by one Ni atom (42% of Archimedean cubes) [12].

Analysis of the interatomic distances in the $ErNi_7B_3$ structure (Table 7) shows that a maximal shortening of distances (9%) is observed between the Er and Ni4

atoms. The other interatomic distances are close to the respective sum of the atomic radii of the components. Because of the presence of such compression between Er and Ni4 atoms, the borides of $ErNi_7B_3$-type structure are formed only with Er, Tm, Yb, Lu, and Sc, i.e., rare earth metals with the smallest atomic radii. In the structures of the corresponding compounds formed by RE metals with the larger atomic radii the compression between RE and Ni atoms will increase. So, these compounds are not formed.

4. Conclusions

The crystal structure $Er_{0.917}Ni_{0.409}B$ and $ErNi_7B_3$ borides have been determined using X-ray single-crystal methods and the relationship between these structures and the other structure types ternary borides, which contain rare-earth metals, has been discussed.

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