Crystal structure of tetraholmium nickel tetradecaboride, Ho_4NiB_{14}

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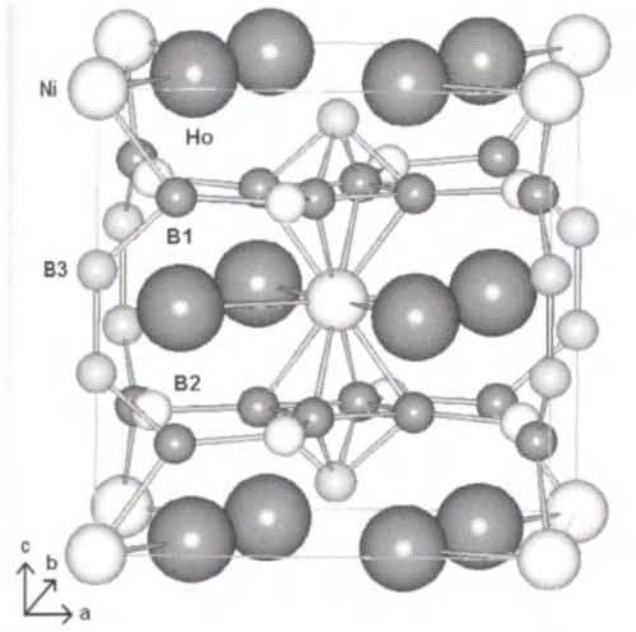

Abstract

$B_{14}Ho_4Ni$, tetragonal, *P4/mnc* (No. 128), $a = 7.2097(8)$ Å, $c = 7.4587(9)$ Å, $V = 387.7$ Å^3, $Z = 2$, $R_{gt}(F) = 0.049$, $wR_{ref}(F^2) = 0.087$, $T = 300$ K.

Source of material

A single-crystalline piece of the title compound has been extracted from a coarse-grained ternary alloy with nominal composition $Ho_2Ni_3B_6$. The latter was prepared from a stoichiometric mixture of holmium pieces, nickel powder and boron powder by arc-melting and subsequent annealing (duration 75 h, temperature up to 1733 K) within a resistance furnace under argon atmosphere. Electron probe microanalysis confirmed the presence of a ternary phase with approximate stoichiometry Ho_4NiB_{13} in addition to the main phase $Ho_2Ni_3B_6$ [1].

Experimental details

Atomic coordinates of Ho_4NiB_{14} correspond to the standardized form according to [2]. Any attempts to refine Ni and B atoms anisotropically resulted in an unreasonable large maximum-to-minimum axis ratio for the displacement tensor of Ni, while the displacement ellipsoids of B1 and B2 became non-positive definite.

Discussion

Lattice parameters and atomic coordinates of Ho_4NiB_{14} are closely related to those of the previously studied compound Er_4NiB_{13} [3]. However, the difference Fourier synthesis based of our more extended single-crystal data set ($(\sin\theta_{max})/\lambda = 0.66$) shows unambiguously, that the B3 atom not occupies the Wyckoff site 2*b*(0,0,1/2), but 4*e*(0,0,*z*) with free *z* parameter, resulting in the stoichiometry Ho_4NiB_{14} instead of Ho_4NiB_{13}. The crystal structure of Ho_4NiB_{14} (*P4/mnc – ihgea*) is characterized by an alternate stacking of planar Ho-Ni layers and slightly corrugated B1-B2 layers perpendicular to the *c* axis. The B1–B2 layers are linked via dumbbells of B3 atoms lying above and below the Ho-Ni layers. Each B3 atom is square-pyramidally surrounded by B1 atoms at 1.72 Å as well as Ho atoms at 2.66 Å. The coordination number of Ni atoms is 12. Their coordination polyhedron consists of a square plane of Ho atoms (Ni—Ho distance 2.770 Å) and two square pyramids of B1 atoms (Ni—B1 distance 2.17 Å).

Table 1. Data collection and handling.

Crystal:	metallic block, size 0.020 × 0.040 × 0.080 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	426.42 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, 186 frames, $\Delta\phi = 1°$
$2\theta_{max}$:	55.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2930, 246
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 209
$N(param)_{refined}$:	17
Programs:	SHELXS-97 [4], SHELXL-97 [5], XABS2 [6], SCHAKAL 97 [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
Ni	2*a*	0	0	0	0.003(1)
B(1)	16*i*	0.175(3)	0.042(2)	0.234(2)	0.006(3)
B(2)	8*g*	0.089(3)	1/2 + *x*	1/4	0.008(4)
B(3)	4*e*	0	0	0.386(6)	0.011(7)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ho	8*h*	0.1939(1)	0.3317(1)	0	0.0052(6)	U_{11}	0.0042(5)	–0.0004(4)	0	0

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