Refinement of the crystal structure of holmium tetranickel boride, $HoNi_4B$

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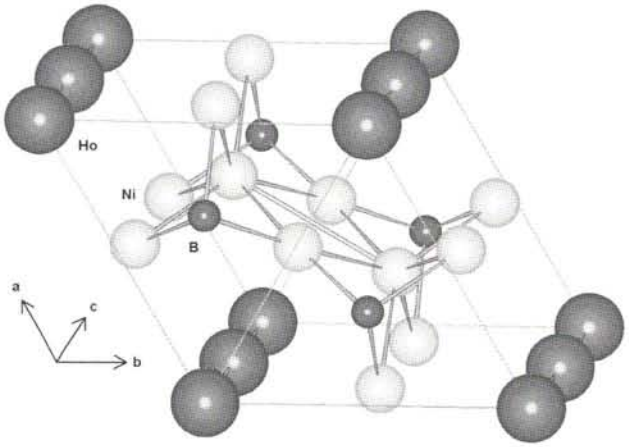

Abstract

$BHoNi_4$, hexagonal, *P6/mmm* (No. 191), $a = 4.9696(4)$ Å, $c = 6.9419(5)$ Å, $V = 148.5$ Å^3, $Z = 2$, $\rho_m = 9.13(1)$ g·cm^{-3}, $R(P) = 0.072$, $wR(P) = 0.099$, $R(I) = 0.065$, $T = 300$ K.

Source of material

A polycrystalline rod with nominal composition $HoNi_4B$ was prepared from holmium pieces, nickel powder and boron powder. A stoichiometric mixture of the high-purity elements was pressed to pellets, which have been arc-melted under argon atmosphere on a water-cooled copper heart. Melting of the buttons has been repeated three times in order to improve the sample homogeneity. The melt was cast into a rod-shaped copper mould. A special annealing treatment (duration 75 h, temperature up to 1370 K) was performed within a resistance furnace under argon atmosphere. The composition of the annealed sample was determined with electron probe microanalysis applying the WDX mode.

Experimental details

Accurate lattice parameters of $HoNi_4B$ were measured at temperatures between 300 K and 25 K using a SIEMENS D5000 powder diffractometer equipped with a low-temperature chamber (Fa. Anton Paar).

Initial values for the refinement of lattice parameters and atomic coordinates were taken from $CaNi_4B$ [2]. Correction of systematic errors of measured peak positions was done by means of refinement parameters displacement and transparency. Refinement of individual isotropic temperature factors failed because of unreasonable parameter shifts. Therefore, an overall temperature factor was refined only.

Discussion

The title compound is isotypic with YNi_4B [1] and $CaNi_4B$ [2]. It was previously studied with powder diffraction, but only lattice parameters were reported [3]. Here we present the first structure refinement of $HoNi_4B$ using X-ray powder diffraction data. $HoNi_4B$ (*P6/mmm* - idcba) crystallizes with the $CeCo_4B$ structure type [4]. Perpendicular to the *c* axis, two layers of $CaCu_5$-type and $CeCo_3B_2$-type are alternately stacked. Parallel to the *c* axis, the Ho atoms form linear chains which are located at channels with hexagonal cross-section (Ho1—Ho2 distance 3.471 Å). Each Ho1 atom is coordinated by 6 Ni2 atoms at 2.862 Å and 12 Ni1 atoms at 3.202 Å, whereas each Ho2 atom is surrounded by 6 B atoms at 2.862 Å and 12 Ni1 atoms at 2.878 Å. The B atoms occupy the centres of trigonal prisms formed by 6 Ni1 atoms (Ni1—B distance 2.041 Å). Atomic coordinates correspond to the standardized form according to STRUCTURE TIDY [5]. The linear thermal expansion at temperatures between 300 K and 25 K indicates a nearly isotropic behaviour: $\alpha_a = 1.3 \times 10^{-5}$ K^{-1}, $\alpha_c = 1.0 \times 10^{-5}$ K^{-1}.

Table 1. Data collection and handling.

Powder:	grey
Wavelength:	Cu K_α radiation (1.54439 Å)
μ:	711.0 cm^{-1}
Diffractometer:	SIEMENS D5000
Scan mode:	Step scan
$2\theta_{max}$, stepwidth:	120°, 0.01
$N(points)_{measured}$:	10501
$N(hkl)_{measured}$:	68
$N(param)_{refined}$:	17
Programs:	WYRIET [6], SCHAKAL92 [7]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
Ni(1)	6*i*	1/2	0	0.2091(1)	0.0082(3)
Ni(2)	2*d*	1/3	2/3	1/2	0.0082
B	2*c*	1/3	2/3	0	0.0082
Ho(1)	1*b*	0	0	1/2	0.0082
Ho(2)	1*a*	0	0	0	0.0082

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