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Refinement of the crystal structure of holmium nickel borocarbide, HoNiBC

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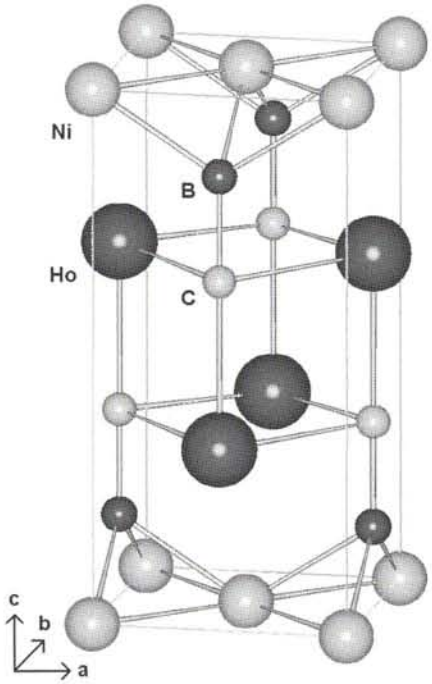

Abstract

BCHoNi, tetragonal, *P4/nmm* (No. 129), $a = 3.5621(5)$ Å, $c = 7.556(2)$ Å, $V = 95.9$ Å^3, $Z = 2$, $R_{gt}(F) = 0.030$, $wR_{ref}(F^2) = 0.076$, $T = 300$ K.

Source of material

A coarse-grained sample with nominal composition HoNiBC was prepared from holmium pieces and powders of nickel, boron and carbon, respectively. 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. A special annealing treatment (duration 75 h, temperature up to 1733 K) was performed in a resistance furnace under argon atmosphere. The composition of the annealed sample was determined with electron probe microanalysis applying the WDX mode. A single grain for single-crystal diffractometry was extracted from the polycrystalline aggregate.

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Experimental details

Accurate lattice parameters of HoNiBC were measured at temperatures between 300 K and 100 K using a STOE STADI4 four-circle diffractometer equipped with a CRYOSTREAM cooling system (Oxford Cryosystems).

Discussion

The title compound is isotypic with LuNiBC [1]. Along the *c* axis, inverse PbO-type Ni_2B_2 layers are separated by double NaCl-type HoC layers, in contrast to the alternate stacking of Ni_2B_2 and HoC layers in the superconducting quaternary phase $HoNi_2B_2C$. This structural modification changes the space group from *I4/mmm* - edba ($HoNi_2B_2C$) to *P4/nmm* - c_3a (HoNiBC). The only known structure refinement on HoNiBC has been performed using X-ray powder diffraction data from a sample containing unidentified impurity phases [2]. Here we present the first structure refinement using single-crystal diffraction data.
Atomic coordinates correspond to the standardized form according to STRUCTURE TIDY [4], shifting the origin by [0 0 1/2]. Compared with LuNiBC, a replacement of the smaller Lu atom by the larger Ho atom shifts the lattice parameters in an opposite manner: the *a* axis expands, whereas the *c* axis contracts. Each Ho atom is in a square-planar coordination by four C atoms at 2.520 Å and vice versa. The Ni atom is tetrahedrally surrounded by four B atoms, with B–Ni–B bond angles of 106.3° and 116.0° and a Ni—B distance of 2.10 Å. The refinement of site occupancies results in the composition $HoNi_{0.97(1)}BC$. The analysis of the U_{ij} reveals a preferred displacement of Ho and Ni atoms in the *a-b* plane. As distinct from $HoNi_2B_2C$ [3], the linear thermal expansion at temperatures between 300 K and 100 K indicates a nearly isotropic behaviour: $\alpha_a = \alpha_c = 1.2 \times 10^{-5}$ K^{-1}.

Table 1. Data collection and handling.

Crystal:	metallic lustre, block, size 0.02 × 0.04 × 0.06 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	502.52 cm^{-1}
Diffractometer, scan mode:	STOE STADI4, ω/θ
$2\theta_{max}$:	74.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1136, 185
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 156
$N(param)_{refined}$:	12
Programs:	STRUCTURE TIDY [4], SHELX-97 [5], SCHAKAL92 [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2), origin at (2/*m*).

Atom	Site	*x*	*y*	*z*	U_{iso}
B	2*c*	1/4	1/4	0.147(2)	0.007(2)
C	2*c*	1/4	1/4	0.345(2)	0.006(2)

Table 3. Atomic coordinates and displacement parameters (in Å^2), origin at (2/*m*).

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ho	2*c*		1/4	1/4	0.6660(1)	0.0064(3)	U_{11}	0.0032(3)	0	0	0
Ni	2*a*	0.97(1)	3/4	1/4	0	0.0068(6)	U_{11}	0.0025(7)	0	0	0

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