Crystal structure of dysprosium borosilicide, $Dy_{0.7}B_{12.33}Si_3$

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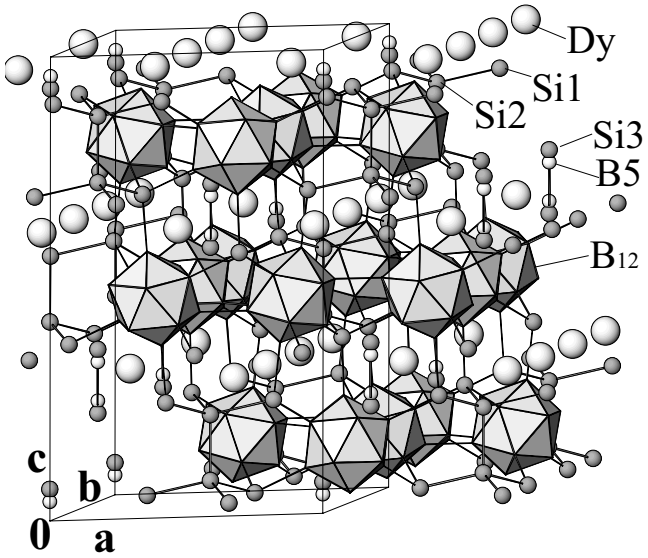

Abstract

$B_{12.33}Dy_{0.7}Si_3$, trigonal, $R\bar{3}m$ (No. 166), a = 10.0782(3) Å, c = 16.4651(4) Å, V = 1448.3 Å^3, Z = 9, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.088, T = 293 K.

Source of material

$Dy_{0.7}B_{12.33}Si_3$ single crystal was grown by using high temperature solution method with silicon as the flux. The starting mixture materials of DyB_4, amophous B, SiB_6 and silicon with nominal composition of $DyB_{10–20}Si_{50}$ was pressed into a pellet, then heated in a BN crucible with a graphite susceptor in a RF furnace. The mixture was first heated to about 1923 K in flowing argon atmosphere, kept there for 8 to 12 hours, then cooled slowly (about 40 K/hour) down to 1473 K. Finally, the power was switched off and the melt was cooled down to room temperature. The obtained ingot was put in HF/HNO_3 solution in order to remove the silicon matrix. The residuals are the rare earth boron-rich single crystals with black colour.

Discussion

The $Dy_{0.7}B_{12.33}Si_3$ compound is isostructural with the newly found compound in Y–B–Si system [1]. The boron atoms of B1 to B4 form the B_{12} icosahedral unit. However, the boron icosahedra are only interconnected into a two-dimensional Kagome lattice. The boron atoms in different layers are not bonded directly. Three boron atoms in three icosahedra, in which two are in one layer and another one in the neighbour layer bond with Si1 atoms which are further linked with Si2—Si2 bridges. Atoms of Si3 and B5 are, in fact, in near split positions both with occupancy of 0.500(7) and U_{iso} of 0.031(3) Å^2 (reset). Dy atoms are sandwitched between two neighbor boron layers with occupancy of 0.697(2).

Table 1. Data collection and handling.

Crystal:	black block, size 0.36 × 0.46 × 0.55 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	85.94 cm^{-1}
Diffractometer, scan mode:	Brucker SMART CCD, ω
$2\theta_{max}$:	103.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	18513, 2022
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2009
$N(param)_{refined}$:	32
Programs:	SIR97 [2], SHELXL-97 [3]

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

Atom	Site	Occ.	x	y	z	U_{iso}
Si(3)	6*c*	0.500(7)	2/3	1/3	0.2909(1)	0.0031(3)
B(1)	36*i*		0.6640(1)	0.1556(1)	0.13538(7)	0.0050(2)
B(2)	36*i*		0.2998(2)	0.0062(2)	0.11530(8)	0.0053(2)
B(3)	18*h*		0.8117(1)	–x	0.0636(1)	0.0057(2)
B(4)	18*h*		0.7580(1)	–x	0.2306(1)	0.0082(3)
B(5)	6*c*	0.500	2/3	1/3	0.2642(4)	0.0031

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References

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2. Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R.: SIR97: A New Tool for Crystal Structure Determination and Refinement. J. Appl. Crystallogr. **32** (1999) 115-119.
3. Sheldrick, G. M.: SHELXL-97: Programm for the refinement of crystal structures. University of Göttingen, Germany 1997.

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Dy(1)	9*e*	0.697(2)	1/2	0	0	0.01191(6)	0.01374(8)	0.00630(6)	$U_{22}/2$	–0.00167(2)	$2U_{13}$
Si(1)	18*h*		0.20195(3)	–x	0.06022(3)	0.0077(2)	U_{11}	0.0043(2)	0.0049(2)	0.00017(6)	–U_{13}
Si(2)	6*c*		1/3	2/3	0.09551(6)	0.0070(2)	U_{11}	0.0050(3)	$U_{11}/2$	0	0

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