Crystal structure of scandium borocarbide, $ScB_{13}C$

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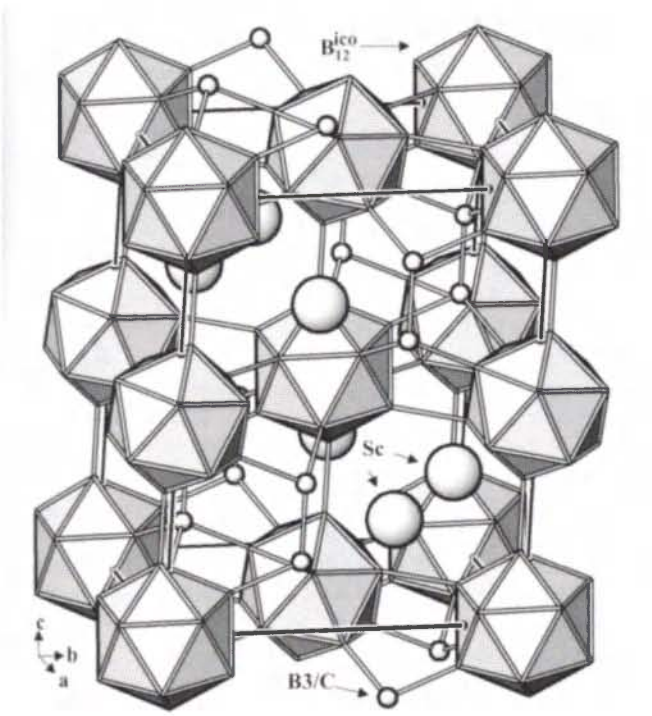

Abstract

$B_{13}CSc$, orthorhombic, *Imam* (No. 74), $a = 5.6829(2)$ Å, $b = 8.0375(3)$ Å, $c = 10.0488(4)$ Å, $V = 459.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.022$, $wR_{ref}(F^2) = 0.059$, $T = 293$ K.

Source of material

Bulk material was produced by sintering appropriate mixtures of ScB_{12}, B and graphite powder at 1973 K in an inductively heated carbon susceptor (experimental details, source of materials as well as details of chemical characterization are given in [1]). Crystal growth was performed in an auxiliary Cu flux. A powder with nominal $ScB_{15}C$ composition (0.5 g) was mixed with about 10 g copper powder, cold pressed into cylindrical shape and inductively heated in a BN crucible placed in a graphite susceptor to about 1873 K under Ar gas. The material was kept at that temperature for 8 hours and slowly cooled (50 K/hour) to 1373 K. Then the power was switched off and the system cooled down to room temperature. The Cu matrix was dissolved in conc. HNO_3. Together with columnar crystals of $ScB_{17}C_{0.25}$ [1] a very few small silvery prismatic crystals of $ScB_{14-y}C_y$ ($y = 1.1$) were found. This refractory compound is stable in mineral acids and air.

Discussion

$ScB_{14-y}C_y$ ($y = 1.1$) adopts the defect $MgAlB_{14}$ [2,3] type of structure. Boron atoms (B1, B2, B4 and B5) form a three dimensional framework thus building up one type of B_{12} icosahedral unit. These B_{12} icosahedra are interconnected by direct *inter*-icosahedral bonds and by one type of boron atom (B3) which acts as a bridge between the icosahedra and is in case of $ScB_{14-y}C_y$ ($y = 1.1$) partially substituted by C (55%). Large interstitial voids in this arrangement are occupied (96%) by Sc metal atoms. Differently to $MgAlB_{14}$, the Al 4*c* position remains unoccupied in case of $ScB_{14-y}C_y$ ($y = 1.1$).

The bond lengths between boron atoms are observed in the range of 1.67 Å < d_{B-B} < 1.86 Å. $ScB_{14-y}C_y$ ($y = 1.1$) is a ternary phase stabilized by small amounts of carbon. The refinement of occupancy parameters based on the present assignment of a mixed occupation of the non icosahedral boron B3 position (indicated by an increased electron density) gave a composition compatible with those obtained by chemical analysis of samples produced by powder metallurgical techniques [1]. Nevertheless the amount of incorporated Sc and C seems to depend on experimental conditions.

Table 1. Data collection and handling.

Crystal:	silvery metallic parallelepiped, size 0.14 × 0.14 × 0.18 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.54 cm^{-1}
Diffractometer, scan mode:	Enraf Nonius CAD4, ω
$2\theta_{max}$:	69.88°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2296, 574
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 516
$N(param)_{refined}$:	40
Programs:	SHELXL-97 [4], ATOMS [5]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
B(3)	8*h*	0.0	0.3685(2)	0.1605(1)	0.0074(3)
C(3)	8*h*	0.0	0.3685	0.1605	0.0074

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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}
Sc	4*e*	0.964(4)	0	0.62133(5)	1/4	0.0098(2)	0.0040(2)	0.0148(2)	0	0	0
B(1)	16*j*		0.1608(2)	0.8392(1)	0.06141(9)	0.0056(4)	0.0046(3)	0.0047(4)	0.0001(3)	0.0000(3)	-0.0002(3)
B(2)	16*j*		0.2430(2)	0.4508(1)	0.08375(9)	0.0051(4)	0.0056(3)	0.0048(4)	0.0003(3)	0.0005(3)	0.0000(3)
B(4)	8*h*		0	0.9742(2)	0.1670(1)	0.0055(5)	0.0051(5)	0.0042(5)	0	0	0.0002(4)
B(5)	8*h*		0	0.8224(2)	0.5949(1)	0.0052(5)	0.0067(5)	0.0037(5)	0	0	0.0003(4)

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