Crystal structure of lanthanum borocarbide, $La_5B_4C_{5-x}$ (x = 0.15)

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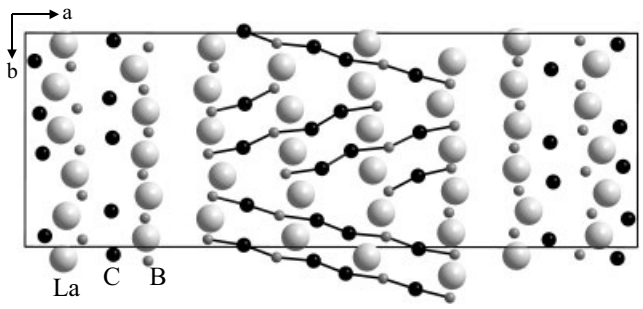

Abstract

$B_8C_{9.71}La_{10}$, orthorhombic, $Pna2_1$ (No. 33), a = 24.657(1) Å, b = 8.6051(5) Å, c = 8.6540(7) Å, V = 1836.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.039, $wR_{ref}(F^2)$ = 0.106, T = 293 K.

Source of material

Cold-pressed pellets of different mixtures of powders prepared from commercially available pure La, B and C were arc melted under argon atmosphere. The samples were then wrapped in molybdenum foil and annealed in silica tubes under argon for 1 week at 1273 K. Shiny black plate-like single crystals were isolated from crushed molten samples. All handling was carried out under argon atmosphere inside a glove box or through the Schlenk technique.

Discussion

The title structure is isotypic to that of $Ce_5B_4C_5$ [1,2]. However, the structure determination concludes slightly different composition with a carbon deficit. The positions of the carbon atoms in the unit BC_2 are not fully occupied. The structure of $La_5B_4C_{5-x}$ (x = 0.15) is composed of slightly corrugated two-dimensional metal atom square nets hosting the finite boron-carbon units B_4C_4, B_3C_3, BC_2 and isolated carbon atoms. Interatomic distances in the unit BC_2 are indicative of double bonds: d(C6—B6) = 1.47(1) Å and d(C4—B6) = 1.43(1) Å, angle ∠C6–B6–C4 = 164.1(9)°. The shortest distance of 1.58(1) Å between B atoms is observed in the unit B_3C_3 for B3—B8. No interaction between carbon atoms was found in the structure $La_5B_4C_{5-x}$. The distances d(La—La) range from 3.4595(6) Å to 3.8754(6) Å, d(La—B) from 2.69(1) Å to 3.110(8) Å, and d(La—C) from 2.495(7) Å to 2.744(7) Å.

Table 1. Data collection and handling.

Crystal:	shiny black platelet, size 0.04 × 0.08 × 0.1 mm
Wavelength:	Ag K_α radiation (0.56086 Å)
μ:	119.84 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, 300 exposures, $\Delta\omega$ = 0.6
$2\theta_{max}$:	56.32°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	53651, 9045
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 8332
$N(param)_{refined}$:	252
Programs:	SIR97 [3], SHELXL-97 [4], DIAMOND [5]

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

Atom	Site	Occ.	x	y	z	U_{iso}
C(4)	4*a*	0.87(4)	0.3049(3)	0.365(1)	0.109(1)	0.013(2)
C(6)	4*a*	0.84(4)	0.4065(4)	0.258(1)	0.986(1)	0.015(2)

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}
La(1)	4*a*	0.80396(2)	0.43239(5)	–0.00110(5)	0.0086(1)	0.0057(2)	0.0152(2)	0.0007(1)	0.0013(1)	0.0012(1)
La(2)	4*a*	0.80229(2)	0.04192(5)	0.79243(4)	0.0089(2)	0.0119(2)	0.0092(2)	–0.0011(1)	0.0002(1)	–0.0016(1)
La(3)	4*a*	0.79808(2)	0.23799(5)	0.40902(5)	0.0100(2)	0.0060(2)	0.0143(2)	–0.0004(1)	–0.0005(1)	0.0016(1)
La(4)	4*a*	0.82234(1)	0.83141(4)	0.20127(4)	0.0105(1)	0.0059(1)	0.0090(1)	–0.0006(1)	0.0001(1)	–0.0001(1)
La(5)	4*a*	0.80280(2)	0.63322(5)	0.59952(5)	0.0180(2)	0.0109(2)	0.0092(1)	0.0027(1)	–0.0010(1)	–0.0012(1)
La(6)	4*a*	0.56299(2)	0.44602(4)	0.40486(4)	0.0094(2)	0.0066(2)	0.0087(2)	0.0003(1)	–0.00010(1)	0.0003(1)
La(7)	4*a*	0.55851(1)	0.04640(5)	0.59372(5)	0.0086(1)	0.0083(2)	0.0079(1)	–0.0015(1)	–0.0005(1)	0.0009(1)
La(8)	4*a*	0.58140(2)	0.83996(5)	0.19674(5)	0.0168(2)	0.0057(1)	0.0102(1)	–0.0004(1)	–0.0006(1)	–0.0001(1)
La(9)	4*a*	0.54959(2)	0.24642(5)	0.98910(5)	0.0195(2)	0.0051(2)	0.0089(2)	–0.0013(1)	0.0008(1)	0.0001(1)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
La(10)	4*a*	0.56745(2)	0.64313(5)	0.78732(5)	0.0112(2)	0.0063(2)	0.0098(1)	0.0002(1)	0.0005(1)	–0.0001(1)
C(1)	4*a*	0.4060(3)	0.4661(8)	0.5880(8)	0.019(3)	0.009(3)	0.006(2)	0.002(2)	–0.003(2)	0.002(2)
C(2)	4*a*	0.4107(3)	0.0476(8)	0.3835(7)	0.011(2)	0.014(3)	0.004(2)	0.002(2)	0.001(2)	–0.002(2)
C(3)	4*a*	0.2012(3)	0.4651(8)	0.8030(9)	0.010(2)	0.007(3)	0.012(2)	–0.002(2)	–0.001(2)	0.003(2)
C(5)	4*a*	0.0847(3)	0.3542(9)	0.3000(8)	0.011(2)	0.011(3)	0.014(3)	0.006(2)	–0.002(2)	–0.003(2)
C(7)	4*a*	0.2011(3)	0.0664(9)	0.9968(8)	0.012(2)	0.009(3)	0.008(2)	–0.002(2)	–0.002(2)	0.002(2)
C(8)	4*a*	0.1939(3)	0.2627(9)	0.3970(8)	0.013(2)	0.007(3)	0.012(2)	0.004(2)	0.002(2)	0.001(2)
C(9)	4*a*	0.0746(3)	0.1572(8)	0.6952(9)	0.012(2)	0.007(2)	0.012(2)	0.001(1)	0.002(2)	0.002(2)
C(10)	4*a*	0.3077(5)	0.162(1)	0.695(1)	0.037(5)	0.016(4)	0.021(3)	0.000(3)	–0.006(4)	–0.001(3)
B(1)	4*a*	0.5287(3)	0.9367(8)	0.8897(8)	0.014(3)	0.004(3)	0.008(3)	–0.001(2)	–0.003(2)	–0.001(2)
B(2)	4*a*	0.1436(3)	0.037(1)	1.0215(9)	0.013(3)	0.011(3)	0.009(3)	0.003(2)	–0.007(2)	0.000(2)
B(3)	4*a*	0.9671(3)	0.0518(9)	0.5981(9)	0.013(3)	0.007(3)	0.010(3)	–0.005(2)	0.000(2)	0.000(2)
B(4)	4*a*	0.0238(3)	0.3746(9)	0.2924(9)	0.011(3)	0.008(3)	0.010(3)	0.002(2)	0.001(2)	0.000(2)
B(5)	4*a*	0.1422(3)	0.492(1)	0.8283(9)	0.005(2)	0.013(3)	0.016(3)	0.000(2)	–0.001(2)	0.001(2)
B(6)	4*a*	0.3584(5)	0.3330(1)	0.053(1)	0.022(5)	0.016(4)	0.015(3)	–0.001(3)	–0.004(3)	0.007(3)
B(7)	4*a*	0.1376(4)	0.3040(1)	0.368(1)	0.014(3)	0.009(3)	0.019(3)	–0.002(2)	0.005(2)	–0.006(2)
B(8)	4*a*	0.5152(3)	0.3669(8)	0.6886(9)	0.011(3)	0.005(3)	0.012(3)	–0.007(2)	0.001(2)	0.000(2)

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