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Orthorhombic Yb_5Si_4 from synchrotron powder data

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Key indicators

Powder synchrotron study
T = 293 K
Mean σ(Si–Si) = 0.016 Å
R factor = 0.074
wR factor = 0.096
Data-to-parameter ratio = 0.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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Orthorhombic Yb_5Si_4 (pentaytterbium tetrasilicide) crystallizes with the structure type Sm_5Ge_4. However, the two compounds differ in the coordination of some Si (Ge) atoms. Each Si atom in the structure of Yb_5Si_4 forms one covalent bond with another Si atom (Si—Si distances of 2.45–2.47 Å), in contrast to Sm_5Ge_4, where only half of the Ge atoms are covalently bonded to another Ge atom (Ge—Ge distance of 2.658 Å).

Comment

During studies of the electronic properties of phases from the Yb–Si system (Alami-Yadri, 1997), a new compound, Yb_5Si_4, was synthesized, and the structure-type Sm_5Ge_4 (Smith *et al.*, 1967) was identified from a comparison of lattice parameters and observed (Guinier films) and calculated (Sm_5Ge_4 structure type) intensities. This compound was reported by Palenzona *et al.* (2002) as novel in the Yb–Si system. However, no refined atomic parameters were given. Our Rietveld refinement using synchrotron data (Fig. 1) confirms that Yb_5Si_4 is isopointal with Sm_5Ge_4.

The structure can be best described as a stacking of three types of (0y0) slabs: slab *A* (Fig. 2) at y = 0.25, 0.75 is flat and is formed from a 3^2434 net of Si atoms centred by a 4^4 net of Yb atoms. Slab *B* (Fig. 3) at $y \sim 0$, 0.5 is puckered and is formed from a 6^3 net of Si atoms. Slab *C* (Fig. 4) at $y \sim 0.10$, 0.40, 0.60, 0.90 is puckered and is formed from a 3^2434 net of Yb atoms, similar to the net of Si atoms found in slab *A*.

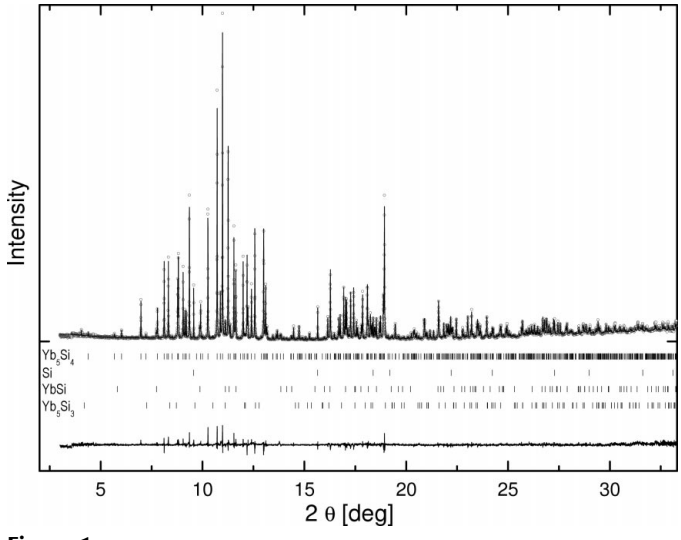

Figure 1
Observed (circles) and calculated (solid line) intensities for Yb_5Si_4. Ticks indicate from top to bottom the positions of Bragg peaks of the main (Yb_5Si_4) and impurity (Si, YbSi, Yb_5Si_3) phases. The difference pattern appears below.

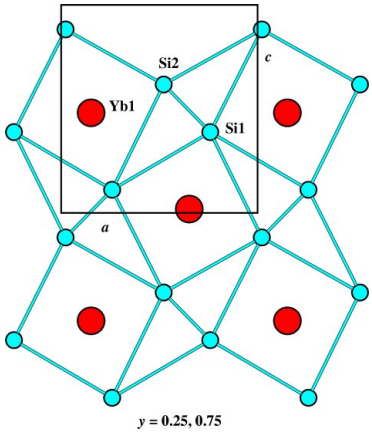

Figure 2
The structural slabs (0y0) of Yb_5Si_4 viewed along the b axis: A at y = 0.25, 0.75.

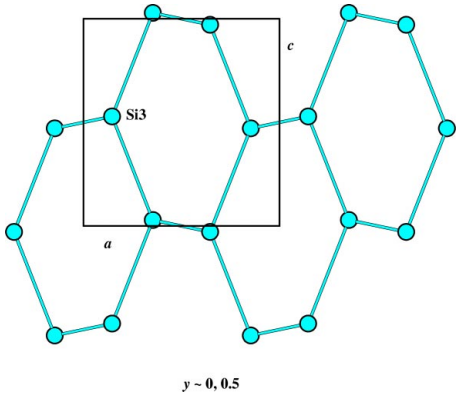

Figure 3
The structural slabs (0y0) of Yb_5Si_4 viewed along the b axis: B at $y \sim 0$, 0.5.

The atoms have similar coordinations to those in Sm_5Ge_4 (Smith *et al.*, 1967), with the exception of Si3, which forms a covalent bond with another Si3 at a distance of 2.454 Å, in contrast to Ge3 in Sm_5Ge_4, where the closest Ge atom (Ge3) is at a distance of 3.706 Å. The same difference between the silicide and the germanide was reported also for the system $Gd_5(Si,Ge)_4$ (Pecharsky & Gschneidner, 1997), and was related to a different magnetic behaviour of the silicide and the germanide.

Experimental

The samples were prepared using a sealed method because of the high vapour pressure of ytterbium. Stoichiometric amounts of the elements were sealed in an evacuated tantalum tube and melted for about one minute by resistance heating.

Crystal data

Yb_5Si_4
M_r = 977.54
Orthorhombic, *Pnma*
a = 7.26327 (4) Å
b = 14.78061 (8) Å
c = 7.70343 (4) Å
V = 827.006 (8) Å^3
Z = 4
D_x = 7.851 (2) Mg m^{-3}
Synchrotron radiation, λ = 0.5230 Å
Cell parameters from 597 reflections
θ = 3.6–33.3°
μ = 1.83 mm^{-1}
T = 293 K
grey
Specimen shape: cylinder
50 × 0.3 × 0.3 mm
Particle morphology: plate-like

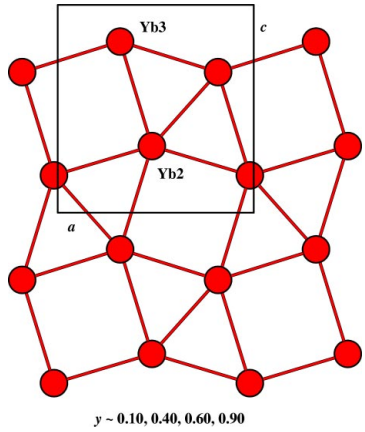

Figure 4
The structural slabs (0y0) of Yb_5Si_4 viewed along the b axis: C at $y \sim 0.10$, 0.40, 0.60, 0.90.

Data collection

2-axis goniometer diffractometer
Specimen mounting: glass capillary
Specimen mounted in transmission mode
Absorption correction: cylindrical
$2\theta_{min}$ = 2.500, $2\theta_{max}$ = 33.333°
Increment in 2θ = 0.005°

Refinement

R_p = 0.074
R_{wp} = 0.096
R_{exp} = 0.075
S = 1.28
$2\theta_{min}$ = 3.600, $2\theta_{max}$ = 33.333°
Increment in 2θ = 0.005°
Wavelength of incident radiation: 0.52301 Å
Profile function: pseudo–Voigt
915 reflections
46 parameters
$(\Delta/\sigma)_{max}$ = 0.01
Preferred orientation correction: none

Table 1
Selected geometric parameters (Å).

Yb1—Si2^{i}	2.887 (16)	Yb2—Yb3^{ix}	3.686 (2)
Yb1—Si1^{ii}	2.939 (15)	Yb2—Yb3^{x}	3.704 (2)
Yb1—Si1	2.953 (15)	Yb2—Yb2^{xi}	3.790 (2)
Yb1—Si3^{iii}	3.147 (9)	Yb2—Yb3^{viii}	3.861 (2)
Yb1—Si2^{iv}	3.229 (15)	Yb2—Yb2^{xii}	3.937 (2)
Yb1—Yb3^{v}	3.425 (3)	Yb3—Si3^{xiii}	2.861 (10)
Yb1—Yb3^{vi}	3.462 (3)	Yb3—Si3	2.871 (11)
Yb1—Yb2^{ii}	3.501 (2)	Yb3—Si2^{xiv}	2.879 (12)
Yb1—Yb2	3.538 (2)	Yb3—Si2^{xv}	2.898 (12)
Yb2—Si3^{vi}	3.020 (12)	Yb3—Si3^{xi}	2.918 (11)
Yb2—Si3^{vii}	3.059 (11)	Yb3—Si1^{vi}	2.947 (11)
Yb2—Si1^{iv}	3.114 (10)	Yb3—Yb3^{xvi}	3.7838 (19)
Yb2—Si3^{viii}	3.123 (11)	Yb3—Yb3^{xvii}	3.804 (2)
Yb2—Si1	3.233 (11)	Si1—Si2^{xviii}	2.47 (2)
Yb2—Si2^{i}	3.263 (10)	Si3—Si3^{xix}	2.454 (16)
Yb2—Si3^{ix}	3.543 (11)		

Symmetry codes: (i) $x-1, y, z-1$; (ii) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}-z$; (iii) $\frac{1}{2}-x, y-\frac{1}{2}, z-\frac{1}{2}$; (iv) $x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}-z$; (v) $1-x, y-\frac{1}{2}, -z$; (vi) $\frac{1}{2}-x, 1-y, z-\frac{1}{2}$; (vii) $-x, 1-y, 1-z$; (viii) $x, y-1, z$; (ix) $x-\frac{1}{2}, y-1, \frac{1}{2}-z$; (x) $-x, 1-y, -z$; (xi) $\frac{1}{2}+x, y, \frac{1}{2}-z$; (xii) $-x, -y, -z$; (xiii) $\frac{1}{2}-x, 2-y, z-\frac{1}{2}$; (xiv) $\frac{3}{2}-x, 1-y, z-\frac{1}{2}$; (xv) $1-x, \frac{1}{2}+y, 1-z$; (xvi) $x, \frac{3}{2}-y, z$; (xvii) $x-\frac{1}{2}, y, \frac{1}{2}-z$; (xviii) $x-\frac{1}{2}, \frac{1}{2}-y, \frac{3}{2}-z$; (xix) $-x, 2-y, 1-z$.

Three impurities were identified in the sample during the Rietveld refinement: Si (9 wt%), YbSi (5 wt%) and Yb_5Si_3 (1 wt%).

Cell refinement: *FULLPROF*99 (Rodríguez-Carvajal, 1999); program(s) used to refine structure: *FULLPROF*99; molecular graphics: *ATOMS* (Dowty, 1993); software used to prepare material for publication: *WinPLOTR* (Roisnel & Rodríguez-Carvajal, 1998) and *ATOMS*.

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