



IUCrData

ISSN 2414-3146

Received 30 October 2023

Accepted 13 December 2023

Edited by S. Bernès, Benemérita Universidad Autónoma de Puebla, México

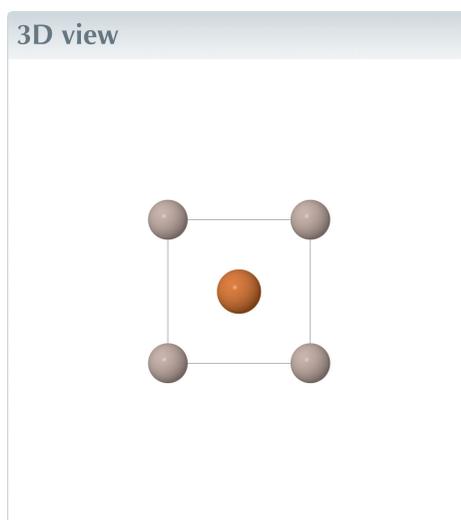
Keywords: crystal structure; B2 phase; Al–Fe system; vacancy.**CCDC reference:** 2314099**Structural data:** full structural data are available from iucrdata.iucr.org

Crystal structure of $\text{AlFe}_{0.95}$

Yibo Liu,^a Huizi Liu,^a Changzeng Fan,^{a,b*} Wen Bin^a and Lifeng Zhang^c

^aState Key Laboratory of Metastable Materials Science and Technology, Yanshan University, Qinhuangdao 066004, People's Republic of China, ^bHebei Key Lab for Optimizing Metal Product Technology and Performance, Yanshan University, Qinhuangdao 066004, People's Republic of China, and ^cSchool of Mechanical and Materials Engineering, North China University of Technology, Beijing, People's Republic of China. *Correspondence e-mail: chzfan@ysu.edu.cn

Three B2-type intermetallic $\text{AlFe}_{1-\delta}$ phases ($0.18 < \delta < 0.05$) in the Al–Fe binary system were synthesized by smelting and high temperature sintering methods. The exact crystal structure for $\delta = 0.05$ was refined by single-crystal X-ray diffraction. The amount of vacancy defects at the Fe atom sites was obtained by refining the corresponding site occupancy factor, converging to the chemical formula $\text{AlFe}_{0.95}$, with a structure identical to that of ideal AlFe models inferred from powder X-ray or neutron diffraction patterns.



Structure description

There are diverse intermetallic phases in the Al–Fe system, among which AlFe has attracted much attention because of its special B2 structure. For example, Van der Kraan & Buschow (1986) studied the crystal structure of the AlFe phase, after heat treatment at 1273 K for 50 h, by X-ray powder diffraction. The authors suggested that AlFe has a CsCl-type structure with cell parameter $a = 2.907 \text{ \AA}$. During the study of the crystal structure of $\text{La}(T,\text{Al})_{13}$ ($T = \text{Fe}, \text{Co}$), a coexisting AlFe cubic phase was discovered. The crystal structure of cubic AlFe was also refined using X-ray powder diffraction data, affording the cell parameter $a = 2.889(4) \text{ \AA}$, and a model in which Al and Fe atoms are occupying the Wyckoff positions $1a$ and $1b$, respectively, in space group $Pm\bar{3}m$ (Guo *et al.*, 1997). Makhlof *et al.* (1994) studied the structure of the magnetic alloys $\text{FeAl}_{1-x}\text{Rh}_x$ by X-ray diffraction, and concluded that the crystal structure of these alloys remains in the B2 structural type. Stein *et al.* (2010), using the high-temperature neutron diffraction approach, found that the cell parameter of the AlFe phase gradually increases by increasing the temperature: the cell parameter of the AlFe phase at room temperature, 373, 1353 and 1393 K, is 2.9097, 2.9136, 2.9681 and 2.9720 \AA , respectively. They also proposed that the AlFe phase has a B2-type crystal structure ($Pm\bar{3}m$ space group, $cP2$ Pearson symbol).



Published under a CC BY 4.0 licence

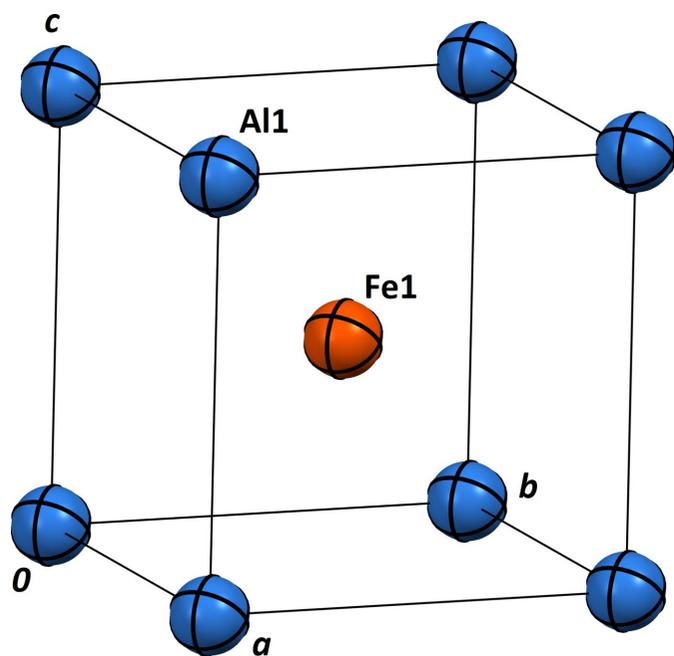


Figure 1
The $\text{AlFe}_{0.95}$ structure (one unit cell), with displacement ellipsoids at the 95% probability level.

In the present work, three kinds of Fe-deficient B2-type $\text{AlFe}_{1-\delta}$ phases were synthesized by smelting and high-temperature sintering methods, with very similar lattice parameters. The $\text{AlFe}_{0.95}$ ($\delta = 0.05$) phase was obtained by the smelting method, while $\text{AlFe}_{0.82}$ and $\text{AlFe}_{0.84}$ phases ($\delta = 0.18$ and $\delta = 0.16$) were obtained from an intergrowth sample by the high-temperature sintering method. The refined chemical formula of the $\text{AlFe}_{0.95}$ phase is in accordance with the complementary EDX results (see Table S1 of the supporting information). Different options for refinements are listed in Table S2 of the supporting information. The structure description reported herein is for the $\text{AlFe}_{0.95}$ ($\delta = 0.05$) phase.

Fig. 1 shows the unit cell of $\text{AlFe}_{0.95}$. The environments of the Al and Fe sites are shown in Figs. 2 and 3, respectively. The Al1 atom at (0, 0, 0) is centred at a rhombic dodecahedron, whose vertices are six Al1 atoms and eight Fe1 atoms;

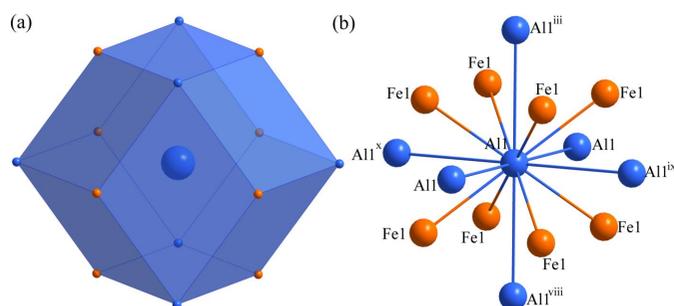


Figure 2
(a) The dodecahedron formed around the Al1 atom at the 1a site and (b) the environment of the Al1 atom with displacement ellipsoids given at the 99% probability level. [Symmetry codes: (iii) $x, y, z + 1$; (viii) $x, y, z - 1$; (ix) $x - 1, y, z$; (x) $x, y - 1, z$.]

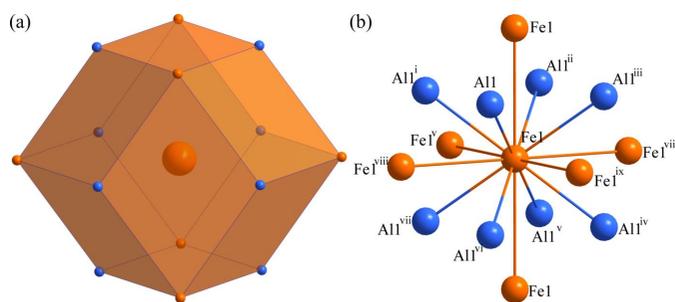


Figure 3
(a) The dodecahedron formed around the Fe1 atom at the 1b site and (b) the environment of the Fe1 atom with displacement ellipsoids given at the 99% probability level. [Symmetry codes: (i) $x + 1, y + 1, z + 1$; (ii) $x + 1, y, z + 1$; (iii) $x, y, z + 1$; (iv) $x + 1, y + 1, z$; (v) $x, y + 1, z$; (vi) $x, y + 1, z + 1$; (vii) $x + 1, y, z$; (viii) $x, y, z - 1$; (ix) $x - 1, y, z$.]

conversely, the Fe1 site at (1/2, 1/2, 1/2) is surrounded by eight Al1 atoms and six Fe1 atoms. The shortest Al1 to Fe1 separation is 2.5164 (4) Å and the shortest Al1 to Al1 link is 2.9057 (5) Å. The R_1 refinement residue versus δ values has been plotted for $0 < \delta < 0.1$ and is shown in Fig. 4, where one can see that R_1 has the lowest value when the chemical occupancy of Fe atoms is 0.95.

Synthesis and crystallization

For the here reported sample obtained by smelting ($\delta = 0.05$), high-purity elements Al (indicated purity 99.95%; 1.629 g) and Fe (indicated purity 99.99%; 3.371 g) were mixed in the stoichiometric ratio 1:1 and the alloy was prepared from the elements by arc melting under an argon atmosphere. Suitable pieces of single-crystal grains were broken and selected from the product for single-crystal X-ray diffraction.

For the sample obtained by high-temperature sintering ($\delta = 0.16$ and 0.18), high-purity elements Al (indicated purity

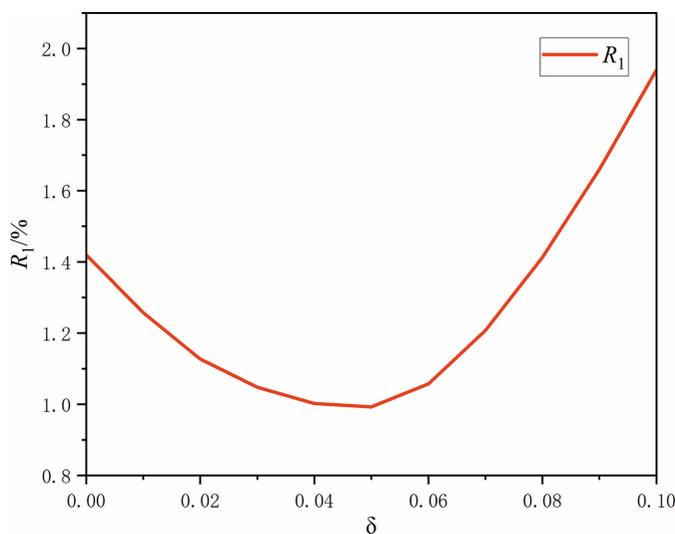


Figure 4
The variation of residual R_1 versus δ for the title compound, obtained by refining the model with different values for δ . The minimum of the curve is at $\delta = 0.05$.

99.95%; 0.7362 g) and Fe (indicated purity 99.9%; 0.2684 g) were mixed in the molar ratio 85:15, ground evenly in an agate mortar, and put into a silicon glass tube, which was vacuum-sealed using a home-made sealing machine. The resulting ampoule was placed in a furnace (SG-XQL1200) and heated up to 473 K for 5 min with a heating rate of 10 K min⁻¹, and then heated up to 1373 K for 2 h with the same heating rate. Finally, the sample was slowly cooled to room temperature by turning off the furnace power. Suitable pieces of single-crystal grains were broken and selected from the product for single-crystal X-ray diffraction.

Refinement

Crystal data, data collection and structure refinement details of AlFe_{0.95} are summarized in Table 1, while crystal data, data collection and structure refinement details of the AlFe_{0.82} and AlFe_{0.84} phases are summarized in Table S3 of the supporting information. Different options for refinement are listed in Table S2. For the AlFe_{0.95} phase, the maximum and minimum residual electron densities in the final difference map are located 1.30 Å and 0.72 Å from Al1.

Funding information

Funding for this research was provided by: The National Natural Science Foundation of China (grant Nos. 52173231 and 51925105); Hebei Natural Science Foundation (grant No. E2022203182); The Innovation Ability Promotion Project of Hebei supported by Hebei Key Lab for Optimizing Metal Product Technology and Performance (grant No. 22567609H).

References

- Brandenburg, K. & Putz, H. (2017). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2015). *APEX3* and *SAINT*. Bruker AXS Inc. Madison, Wisconsin, USA, 2008.
- Guo, Y., Liang, J., Zhang, X., Tang, W., Zhao, Y. & Rao, G. (1997). *J. Alloys Compd.* **257**, 69–74.

Table 1

Experimental details.

Crystal data	
Chemical formula	AlFe _{0.95}
M_r	80.04
Crystal system, space group	Cubic, $Pm\bar{3}m$
Temperature (K)	300
a (Å)	2.9057 (5)
V (Å ³)	24.53 (1)
Z	1
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	14.45
Crystal size (mm)	0.10 × 0.08 × 0.06
Data collection	
Diffractometer	Bruker D8 Venture Photon 100 CMOS
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
T_{\min} , T_{\max}	0.560, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	815, 17, 17
R_{int}	0.032
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.709
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.010, 0.026, 1.44
No. of reflections	17
No. of parameters	3
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.14, -0.24

Computer programs: *APEX3* and *SAINT* (Bruker, 2015), *SHELXT2014/5* (Sheldrick, 2015a), *SHELXL2016/6* (Sheldrick, 2015b), *DIAMOND* (Brandenburg & Putz, 2017), *Mercury* (Macrae *et al.*, 2020) and *pubCIF* (Westrip, 2010).

- Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
- Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
- Makhlouf, S. A., Nakamura, T. & Shiga, M. (1994). *J. Magn. Magn. Mater.* **135**, 257–264.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Stein, F., Vogel, S. C., Eumann, M. & Palm, M. (2010). *Intermetallics*, **18**, 150–156.
- Van der Kraan, A. M. & Buschow, K. H. J. (1986). *Phys. B+C*, **138**, 55–62.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2023). **8**, x231065 [https://doi.org/10.1107/S2414314623010659]

Crystal structure of $\text{AlFe}_{0.95}$

Yibo Liu, Huizi Liu, Changzeng Fan, Wen Bin and Lifeng Zhang

Aluminium iron

Crystal data

$\text{AlFe}_{0.95}$
 $M_r = 80.04$
 Cubic, $Pm\bar{3}m$
 $a = 2.9057$ (5) Å
 $V = 24.53$ (1) Å³
 $Z = 1$
 $F(000) = 38$
 $D_x = 5.417$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 750 reflections
 $\theta = 7.0$ – 30.3°
 $\mu = 14.45$ mm⁻¹
 $T = 300$ K
 Lump, dark gray
 $0.10 \times 0.08 \times 0.06$ mm

Data collection

Bruker D8 Venture Photon 100 CMOS
 diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Krause *et al.*, 2015)
 $T_{\min} = 0.560$, $T_{\max} = 0.746$
 815 measured reflections

17 independent reflections
 17 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$
 $\theta_{\max} = 30.3^\circ$, $\theta_{\min} = 7.0^\circ$
 $h = -4 \rightarrow 4$
 $k = -4 \rightarrow 4$
 $l = -4 \rightarrow 4$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.010$
 $wR(F^2) = 0.026$
 $S = 1.44$
 17 reflections
 3 parameters
 0 restraints

Primary atom site location: structure-invariant
 direct methods
 $w = 1/[\sigma^2(F_o^2) + (0.0141P)^2 + 0.007P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.14$ e Å⁻³
 $\Delta\rho_{\min} = -0.23$ e Å⁻³

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Fe1	0.500000	0.500000	0.500000	0.0084 (3)	0.9499
Al1	0.000000	0.000000	0.000000	0.0086 (4)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0084 (3)	0.0084 (3)	0.0084 (3)	0.000	0.000	0.000
Al1	0.0086 (4)	0.0086 (4)	0.0086 (4)	0.000	0.000	0.000

Geometric parameters (Å, °)

Fe1—Al1 ⁱ	2.5164 (4)	Fe1—Fe1 ^{viii}	2.9057 (5)
Fe1—Al1	2.5164 (4)	Fe1—Fe1 ^v	2.9057 (5)
Fe1—Al1 ⁱⁱ	2.5164 (4)	Fe1—Fe1 ^{vii}	2.9057 (5)
Fe1—Al1 ⁱⁱⁱ	2.5164 (4)	Fe1—Fe1 ^{ix}	2.9057 (5)
Fe1—Al1 ^{iv}	2.5164 (4)	Al1—Al1 ⁱⁱⁱ	2.9057 (5)
Fe1—Al1 ^v	2.5164 (4)	Al1—Al1 ^{viii}	2.9057 (5)
Fe1—Al1 ^{vi}	2.5164 (4)	Al1—Al1 ^x	2.9057 (5)
Fe1—Al1 ^{vii}	2.5164 (4)	Al1—Al1 ^{ix}	2.9057 (5)
Al1 ⁱ —Fe1—Al1	180.0	Fe1 ^{xi} —Al1—Fe1	180.0
Al1 ⁱ —Fe1—Al1 ⁱⁱ	70.5	Fe1 ^{xi} —Al1—Fe1 ^x	109.5
Al1—Fe1—Al1 ⁱⁱ	109.5	Fe1—Al1—Fe1 ^x	70.5
Al1 ⁱ —Fe1—Al1 ⁱⁱⁱ	109.5	Fe1 ^{xi} —Al1—Fe1 ^{xii}	70.5
Al1—Fe1—Al1 ⁱⁱⁱ	70.529 (1)	Fe1—Al1—Fe1 ^{xii}	109.5
Al1 ⁱⁱ —Fe1—Al1 ⁱⁱⁱ	70.5	Fe1 ^x —Al1—Fe1 ^{xii}	70.5
Al1 ⁱ —Fe1—Al1 ^{iv}	70.529 (1)	Fe1 ^{xi} —Al1—Fe1 ^{xiii}	70.5
Al1—Fe1—Al1 ^{iv}	109.471 (1)	Fe1—Al1—Fe1 ^{xiii}	109.5
Al1 ⁱⁱ —Fe1—Al1 ^{iv}	109.5	Fe1 ^x —Al1—Fe1 ^{xiii}	180.0
Al1 ⁱⁱⁱ —Fe1—Al1 ^{iv}	180.0	Fe1 ^{xii} —Al1—Fe1 ^{xiii}	109.5
Al1 ⁱ —Fe1—Al1 ^v	109.5	Fe1 ^{xi} —Al1—Fe1 ^{viii}	109.5
Al1—Fe1—Al1 ^v	70.5	Fe1—Al1—Fe1 ^{viii}	70.5
Al1 ⁱⁱ —Fe1—Al1 ^v	180.0	Fe1 ^x —Al1—Fe1 ^{viii}	109.5
Al1 ⁱⁱⁱ —Fe1—Al1 ^v	109.5	Fe1 ^{xii} —Al1—Fe1 ^{viii}	180.0
Al1 ^{iv} —Fe1—Al1 ^v	70.5	Fe1 ^{xiii} —Al1—Fe1 ^{viii}	70.5
Al1 ⁱ —Fe1—Al1 ^{vi}	70.529 (1)	Fe1 ^{xi} —Al1—Fe1 ^{ix}	109.5
Al1—Fe1—Al1 ^{vi}	109.471 (1)	Fe1—Al1—Fe1 ^{ix}	70.5
Al1 ⁱⁱ —Fe1—Al1 ^{vi}	109.5	Fe1 ^x —Al1—Fe1 ^{ix}	109.5
Al1 ⁱⁱⁱ —Fe1—Al1 ^{vi}	70.5	Fe1 ^{xii} —Al1—Fe1 ^{ix}	70.5
Al1 ^{iv} —Fe1—Al1 ^{vi}	109.5	Fe1 ^{xiii} —Al1—Fe1 ^{ix}	70.5
Al1 ^v —Fe1—Al1 ^{vi}	70.5	Fe1 ^{viii} —Al1—Fe1 ^{ix}	109.5
Al1 ⁱ —Fe1—Al1 ^{vii}	109.5	Fe1 ^{xi} —Al1—Fe1 ^{xiv}	70.5
Al1—Fe1—Al1 ^{vii}	70.5	Fe1—Al1—Fe1 ^{xiv}	109.5
Al1 ⁱⁱ —Fe1—Al1 ^{vii}	70.5	Fe1 ^x —Al1—Fe1 ^{xiv}	70.5
Al1 ⁱⁱⁱ —Fe1—Al1 ^{vii}	109.5	Fe1 ^{xii} —Al1—Fe1 ^{xiv}	109.5
Al1 ^{iv} —Fe1—Al1 ^{vii}	70.5	Fe1 ^{xiii} —Al1—Fe1 ^{xiv}	109.5
Al1 ^v —Fe1—Al1 ^{vii}	109.5	Fe1 ^{viii} —Al1—Fe1 ^{xiv}	70.5
Al1 ^{vi} —Fe1—Al1 ^{vii}	180.0	Fe1 ^{ix} —Al1—Fe1 ^{xiv}	180.0
Al1 ⁱ —Fe1—Fe1 ^{viii}	125.3	Fe1 ^{xi} —Al1—Al1 ⁱⁱⁱ	125.3
Al1—Fe1—Fe1 ^{viii}	54.7	Fe1—Al1—Al1 ⁱⁱⁱ	54.7
Al1 ⁱⁱ —Fe1—Fe1 ^{viii}	125.3	Fe1 ^x —Al1—Al1 ⁱⁱⁱ	54.7
Al1 ⁱⁱⁱ —Fe1—Fe1 ^{viii}	125.3	Fe1 ^{xii} —Al1—Al1 ⁱⁱⁱ	54.7
Al1 ^{iv} —Fe1—Fe1 ^{viii}	54.7	Fe1 ^{xiii} —Al1—Al1 ⁱⁱⁱ	125.3
Al1 ^v —Fe1—Fe1 ^{viii}	54.7	Fe1 ^{viii} —Al1—Al1 ⁱⁱⁱ	125.3
Al1 ^{vi} —Fe1—Fe1 ^{viii}	125.3	Fe1 ^{ix} —Al1—Al1 ⁱⁱⁱ	54.7
Al1 ^{vii} —Fe1—Fe1 ^{viii}	54.7	Fe1 ^{xiv} —Al1—Al1 ⁱⁱⁱ	125.3
Al1 ⁱ —Fe1—Fe1 ^v	54.7	Fe1 ^{xi} —Al1—Al1 ^{viii}	54.7

All—Fe1—Fe1 ^v	125.3	Fe1—All—All ^{viii}	125.3
All ⁱⁱ —Fe1—Fe1 ^v	125.3	Fe1 ^x —All—All ^{viii}	125.3
All ⁱⁱⁱ —Fe1—Fe1 ^v	125.3	Fe1 ^{xii} —All—All ^{viii}	125.3
All ^{iv} —Fe1—Fe1 ^v	54.7	Fe1 ^{xiii} —All—All ^{viii}	54.7
All ^v —Fe1—Fe1 ^v	54.7	Fe1 ^{viii} —All—All ^{viii}	54.7
All ^{vi} —Fe1—Fe1 ^v	54.7	Fe1 ^{ix} —All—All ^{viii}	125.3
All ^{vii} —Fe1—Fe1 ^v	125.3	Fe1 ^{xiv} —All—All ^{viii}	54.7
Fe1 ^{viii} —Fe1—Fe1 ^v	90.0	All ⁱⁱⁱ —All—All ^{viii}	180.0
All ⁱ —Fe1—Fe1 ^{vii}	54.7	Fe1 ^{xi} —All—All ^x	54.7
All—Fe1—Fe1 ^{vii}	125.3	Fe1—All—All ^x	125.3
All ⁱⁱ —Fe1—Fe1 ^{vii}	54.7	Fe1 ^x —All—All ^x	54.7
All ⁱⁱⁱ —Fe1—Fe1 ^{vii}	125.3	Fe1 ^{xii} —All—All ^x	54.7
All ^{iv} —Fe1—Fe1 ^{vii}	54.7	Fe1 ^{xiii} —All—All ^x	125.3
All ^v —Fe1—Fe1 ^{vii}	125.3	Fe1 ^{viii} —All—All ^x	125.3
All ^{vi} —Fe1—Fe1 ^{vii}	125.3	Fe1 ^{ix} —All—All ^x	125.3
All ^{vii} —Fe1—Fe1 ^{vii}	54.7	Fe1 ^{xiv} —All—All ^x	54.7
Fe1 ^{viii} —Fe1—Fe1 ^{vii}	90.0	All ⁱⁱⁱ —All—All ^x	90.0
Fe1 ^v —Fe1—Fe1 ^{vii}	90.0	All ^{viii} —All—All ^x	90.0
All ⁱ —Fe1—Fe1 ^{ix}	125.3	Fe1 ^{xi} —All—All ^{ix}	54.7
All—Fe1—Fe1 ^{ix}	54.7	Fe1—All—All ^{ix}	125.3
All ⁱⁱ —Fe1—Fe1 ^{ix}	125.3	Fe1 ^x —All—All ^{ix}	125.3
All ⁱⁱⁱ —Fe1—Fe1 ^{ix}	54.7	Fe1 ^{xii} —All—All ^{ix}	54.7
All ^{iv} —Fe1—Fe1 ^{ix}	125.3	Fe1 ^{xiii} —All—All ^{ix}	54.7
All ^v —Fe1—Fe1 ^{ix}	54.7	Fe1 ^{viii} —All—All ^{ix}	125.3
All ^{vi} —Fe1—Fe1 ^{ix}	54.7	Fe1 ^{ix} —All—All ^{ix}	54.7
All ^{vii} —Fe1—Fe1 ^{ix}	125.3	Fe1 ^{xiv} —All—All ^{ix}	125.3
Fe1 ^{viii} —Fe1—Fe1 ^{ix}	90.0	All ⁱⁱⁱ —All—All ^{ix}	90.0
Fe1 ^v —Fe1—Fe1 ^{ix}	90.0	All ^{viii} —All—All ^{ix}	90.0
Fe1 ^{vii} —Fe1—Fe1 ^{ix}	180.0	All ^x —All—All ^{ix}	90.0

Symmetry codes: (i) $x+1, y+1, z+1$; (ii) $x+1, y, z+1$; (iii) $x, y, z+1$; (iv) $x+1, y+1, z$; (v) $x, y+1, z$; (vi) $x, y+1, z+1$; (vii) $x+1, y, z$; (viii) $x, y, z-1$; (ix) $x-1, y, z$; (x) $x, y-1, z$; (xi) $x-1, y-1, z-1$; (xii) $x-1, y-1, z$; (xiii) $x-1, y, z-1$; (xiv) $x, y-1, z-1$.