

Linzhiite, FeSi₂, a redefined and revalidated new mineral species from Luobusha, Tibet, China

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Abstract: “Ferdisilicite” (FeSi₂) was first described as a member of the Fe–Si alloy mineral series by Gevork’yan (1969), without CNMMN approval. It is redefined here as a new mineral, revalidated with the new name linzhiite with a CNMNC approval (IMA 2010-011). The type sample was found in a podiform chromitite from the Luobusha ophiolite in Tibet, People’s Republic of China. It occurs as irregular grains ranging from 0.04 to 0.5 mm in diameter and intergrowths with zangboite and native silicon. Linzhiite is steel grey in colour, opaque, with a metallic lustre and a greyish-black streak. The mineral is brittle and has a conchoidal fracture. Cleavage was not observed. The Mohs hardness is 6½, and the calculated density is 4.972 g/cm³. Reflectance values of the material in air (*R*%) are: 26.3 at 470 nm, 30.3 at 546 nm, 32.9 at 589 nm, and 36.3 at 650 nm. The composition (mean of 12 EMP analyses) is Fe 49.09, Si 50.00, Al 0.64 and Mn 0.28 wt%, sum 100.01 wt%. The ideal formula is FeSi₂. The crystal structure was refined from single-crystal X-ray diffraction data to *R* = 0.043. The mineral has a tetragonal *P4/mmm* structure, with *a* = 2.696 (1) Å, *b* = 2.696 (1) Å, *c* = 5.147 (6), *V* = 37.41 (14) Å³, *Z* = 1. The five strongest X-ray powder diffraction lines [*d*-spacing in Å (*hkl*)] are: 5.136 (96), 2.374 (81), 1.896 (55), 1.849 (100), and 1.086 (36). Linzhiite formed in a strongly reducing environment and possibly occurs as xenocrysts derived from mantle sources.

Key-words: linzhiite, new mineral, FeSi₂, iron silicide, crystal structure, chromitite, Luobusha ophiolite, Tibet.

1. Introduction

Gevork’yan *et al.* (1969) reported two new alloy minerals, “*fersilicite*” (FeSi) and “*ferdisilicite*” (FeSi₂), found in heavy-mineral concentrates from placers and drill-core samples in sandstones from the Poltava series, Ukraine. These names have been widely used in the literature (Ostaschenko, 1979; Zhang *et al.*, 1985; Liu *et al.*, 1995; Wartchow *et al.*, 1997; Mahesh *et al.*, 2004) and in mineralogy databases (<http://webmineral.com/>). However, the status of *ferdisilicite* and *fersilicite* as valid species was still controversial, since the species and name were without approval by the IMA-CNMNC.

The studies of natural Si–Fe alloys have been mainly carried out on meteorites and lunar soils. Recently Si–Fe minerals were identified occurring in the podiform ophiolitic chromitites at Luobusha in Tibet (Bai *et al.*, 2002, 2003a, 2004; Robinson *et al.*, 2004). This led to the definition of several new mineral species in the Fe–Si and Ti–Fe–Si series: luobusaite, Fe_{0.83}Si₂ (IMA2005-052a, Bai *et al.*, 2006), zangboite, TiFeSi₂ (IMA2007-036, Li *et al.*, 2009) and naquite, FeSi (IMA2010-010), all approved by the IMA-CNMNC. Among the iron silicide intermetallic compounds observed there, FeSi₂ was the most common.

Intergrowths between FeSi₂, zangboite and native silicon were also observed (Fig. 1). The FeSi₂ phase discovered in Luobusha appears to be identical to the one described as “*ferdisilicite*”. In this paper we revalidate the existence of the FeSi₂ phase as a mineral, describe its properties and redefine it under the name of linzhiite as a new species of Si–Fe alloy mineral discovered in chromitite, Luobusha ophiolite, Tibet. The mineral species and name were approved by the CNMMN (IMA 2010-011). Linzhiite is named for the Linzhi Prefecture, which is situated close to the new type locality. The type specimen of this new mineral has been deposited at the Geological Museum of China (specimen no. M11799) and at the Institute of Geology, Chinese Academy of Geological Sciences, Beijing, China (specimen no. 97-6).

2. Occurrence and mineral separation procedures

The Luobusha podiform chromitites are situated in the Luobusha ophiolite of the Indus–Yarlung Zangbo suture zone, a major tectonic boundary that separates the Lhasa

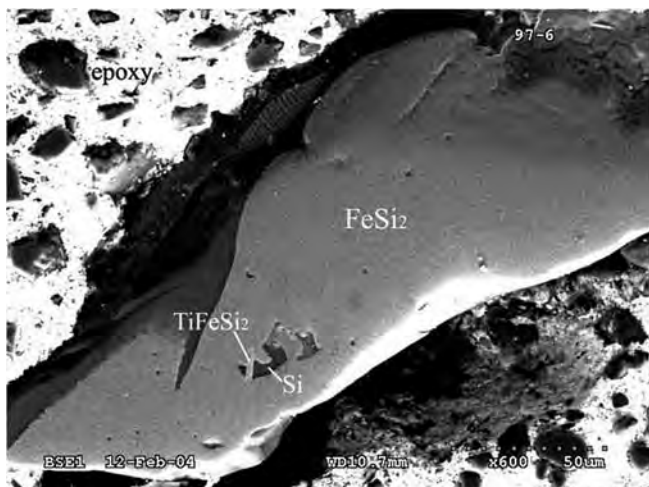


Fig. 1. SEM image of linzhiite (FeSi_2) and its inclusions: zangboite (TiFeSi_2) and silicon (Si).

Block to the north from the Indian continent to the south. The ophiolite extends along Yarlong Zangbo (Brahmaputra) River for about 42 km from east to west and has a width of 1–4 km, with an exposed area of about 70 km². It consists mainly of harzburgite, with lesser amounts of dunite, cumulate mafic rocks, pillow lavas, and an ophiolitic mélange. The chromitites occur as tabular, lenticular and podiform bodies in depleted harzburgites. A wide variety of minerals have been handpicked from the chromitite separates, including diamond, coesite, other native elements, carbides, PGE and base-metal alloys, nitride, silicides, sulphides, silicates and oxides (Bai *et al.*, 2003b; Yang *et al.*, 2007). The linzhiite described here was separated from the heavy-mineral constituents derived from a 1500 kg sample of chromitite collected from ore body 31, Group II of the Luobusha mining district, located about 200 km southeast of Lhasa, in Qusong county, Shannan Prefecture, Tibet (lat. 29°13.50' N, long. 92°12.18' E).

The minerals were hand-picked from this heavy-mineral separate and the possibility of natural or anthropogenic contamination was ruled out. The samples were taken directly from the outcrop and carefully cleaned before processing. All equipment was dismantled and carefully cleaned before crushing and processing (Bai *et al.*, 2000). A 200-kg sample of granite from the Gangdese batholith was processed first to test for contamination during the separation process. Only quartz, feldspar, garnet, mica, apatite, and zircon were recovered from the granite. Any metallic mineral would have been readily recognized but none was found. Using the same procedures and equipment with which the granite sample was processed, we found about 70–80 distinct metallic mineral species and alloys in the samples from the Luobusha chromitite. These minerals include native metals and intermetallic compounds, grains of Os–Ir alloy with inclusions of diamond and metallic carbides (Bai *et al.*, 2003b). However, none of these minerals occur in the granite sample. Similar phases have also been recovered from these chromitites in other laboratories

(Robinson & Yang, 2008; Yamamoto *et al.*, 2009). The samples were hand-washed, air-dried, and crushed carefully to avoid any possible anthropogenic contamination. Mineral separation was carried out using gravity, magnetic and electrical techniques in the Zhengzhou Institute of Multipurpose Utilization of Mineral Resources, Chinese Academy of Geological Sciences. More than a dozen grains of linzhiite were hand-picked from separates of various grain sizes. Each grain was given a number and mounted in epoxy, polished, and analyzed with an electron microprobe. Therefore, we are confident that the Luobusha sample is free of contamination. The FeSi_2 specimens studied are clearly of natural origin.

3. Appearance and physical properties

Linzhiite occurs as grains or tabular crystals generally between 0.04 and 0.5 mm in diameter. The linzhiite colour is steel grey. The streak is greyish-black and the lustre is metallic. Crystals of linzhiite are opaque. The mineral is brittle and has a conchoidal fracture. Cleavage was not observed. The Mohs hardness is 6½, and the calculated density is 4.972 g/cm³. Reflectivity measurements were made in air, using a MPV–3 instrument and WC as a standard. The reflectance values for linzhiite are listed in Table 1.

4. Chemical data

Quantitative chemical analyses were carried out using an electron microprobe (EPMA–1600 Shimadzu), with an accelerating voltage of 15 kV, 15 nA beam current, and a beam diameter of 1 or 5 µm. Multiple element standards from SW9100 NIST were used for calibration (Fe, Si, Al and Mn). The results, based on the average of 12 point analyses of four grains, are given in Table 2.

The typical composition is close to the ideal one. However, Al and Mn were detected in some grains. The empirical formula (based on 3 atoms *pfu*) is $\text{Fe}_{0.98}\text{Al}_{0.03}\text{Mn}_{0.01}\text{Si}_{1.99}$. The simplified formula is FeSi_2 , which has the ideal composition Fe 49.85, Si 50.15 wt%, for a total of 100.00 wt%.

Table 1. Reflectance values for linzhiite (in air).

| <i>R</i> (%) | λ (nm) | <i>R</i> (%) | λ (nm) |
|--------------|----------------|--------------|----------------|
| 23.3 | 400 | 30.9 | 560 |
| 24 | 420 | 32.7 | 580 |
| 27.1 | 440 | 32.9 | 589 (COM) |
| 28 | 460 | 32.9 | 600 |
| 26.3 | 470 (COM) | 35 | 620 |
| 24.6 | 480 | 33.2 | 640 |
| 27.7 | 500 | 36.3 | 650 (COM) |
| 29.8 | 520 | 39.3 | 660 |
| 29.9 | 540 | 39.2 | 680 |
| 30.3 | 546 (COM) | 39 | 700 |

Table 2. Chemical composition of linzhiite (mean of 12 EPM analyses).

| Constituent | Wt% | Range | Probe standard |
|-------------|------------|-------------|----------------|
| Fe | 49.09 (64) | 47.15–51.57 | Fe |
| Si | 50.00 (60) | 49.33–52.85 | Si |
| Al | 0.64 (28) | 0.00–1.87 | Al |
| Mn | 0.28 (11) | 0.00–0.74 | Mn |
| Total | 100.01 | | |

5. X-ray powder diffraction

The powder X-ray diffraction study was performed on a SMART APEX-CCD area-detector diffractometer using MoK α radiation, 45 kV, 35 mA. We used a new method by taking powder-diffraction Debye image with a single crystal grain (Li *et al.*, 2005), 0.08–0.2 mm grains in size and repeat rotation with $\Delta\omega = 10\text{--}20^\circ$. The exposure time was 60–120 s and very clear Debye ring images were obtained. The powder diffraction pattern and diffraction data were obtained using Gadds software; the diffraction pattern is shown in Fig. 2, the data are listed in Table 3 (in Å for MoK α).

The powder X-ray diffraction patterns of linzhiite correspond to a tetragonal FeSi₂ phase. The patterns of the four grains show similar characteristics. The main diffraction peaks [*d*-spacing in Å (*hkl*)] are 1.85 (100), 5.22 (90), 2.37

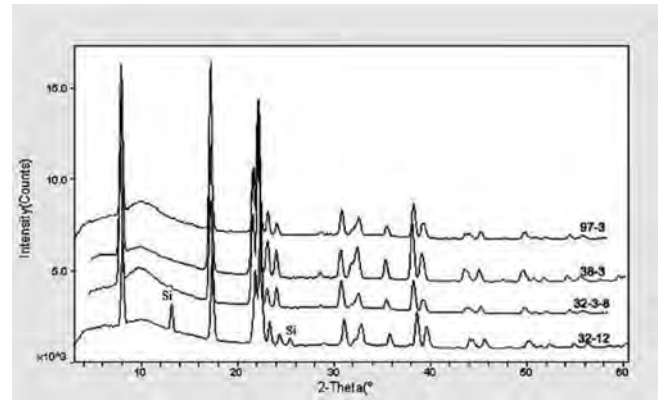


Fig. 2. X-ray powder diffraction pattern of linzhiite (MoK α , $\lambda = 0.71073$ Å), native silicon (Si) is also present (*cf.* Fig. 1).

(80), and 1.89 (80) Å (Table 3). Besides the main mineral linzhiite, samples 32-12 and 32-3-8 were found to contain small inclusions of native Si. Its typical X-ray diffraction peaks are 3.122, 1.894, and 1.629 Å (Fig. 2).

6. The crystal structure of linzhiite

The single-crystal experiments were carried out using a BRUKER SMART CCD diffractometer with 0.1×0.08 mm \times 0.06 mm size of grain (No. 97-3), 2.4 kW

Table 3. X-ray powder diffraction data of linzhiite.

| Number hkl | 32-3-8 | | 38-3 | | 97-3 | | 32-12 | | ICDD73-1843 ^a | |
|---------------|--------------|-------------------------|--------------|-------------------------|--------------|-------------------------|--------------|-------------------------|--------------------------|-------------------------|
| | <i>d</i> (Å) | <i>I</i> _{rel} | <i>d</i> (Å) | <i>I</i> _{rel} | <i>d</i> (Å) | <i>I</i> _{rel} | <i>d</i> (Å) | <i>I</i> _{rel} | <i>d</i> (Å) | <i>I</i> _{rel} |
| 001 | 5.150 | 953 | 5.163 | 918 | 5.142 | 999 | 5.092 | 692 | 5.128 | 999 |
| 010 | | | | | | | | | 2.684 | 8 |
| 002 | | | | | | | | | 2.564 | 3 |
| 011 | 2.373 | 659 | 2.376 | 999 | 2.377 | 697 | 2.372 | 659 | 2.378 | 710 |
| 110 | 1.895 | 609 | 1.899 | 473 | 1.897 | 480 | 1.894 | 468 | 1.897 | 520 |
| 012 | 1.848 | 999 | 1.851 | 793 | 1.851 | 931 | 1.847 | 999 | 1.854 | 925 |
| 111 | 1.776 | 108 | 1.774 | 158 | 1.776 | 167 | 1.773 | 166 | 1.779 | 148 |
| 003 | 1.704 | 129 | 1.708 | 115 | 1.707 | 84 | 1.699 | 76 | 1.709 | 80 |
| 112 | | | | | | | | | 1.525 | 4 |
| 013 | 1.436 | 12 | 1.44 | 21 | 1.439 | 20 | 1.434 | 15 | 1.441 | 30 |
| 020 | 1.34 | 147 | 1.342 | 120 | 1.341 | 170 | 1.339 | 172 | 1.342 | 131 |
| 021 | 1.297 | 13 | 1.299 | 44 | 1.3 | 17 | 1.297 | 28 | 1.298 | 38 |
| 004 | | | | | | | | | 1.282 | 47 |
| 113 | 1.267 | 76 | 1.271 | 104 | 1.269 | 112 | 1.267 | 136 | 1.27 | 92 |
| 120 | | | | | | | | | 1.200 | 3 |
| 022 | | | | | | | | | 1.189 | 2 |
| 121 | 1.167 | 58 | 1.169 | 84 | 1.169 | 76 | 1.167 | 92 | 1.168 | 83 |
| 014 | | | | | | | | | 1.156 | 2 |
| 122 | 1.086 | 193 | 1.087 | 246 | 1.088 | 260 | 1.085 | 251 | | |
| 114 | 1.061 | 75 | 1.061 | 106 | 1.063 | 116 | 1.059 | 146 | | |
| 015 | 0.957 | 32 | 0.958 | 52 | 0.959 | 54 | 0.956 | 47 | | |
| 220 | | | | | 0.949 | 41 | 0.948 | 27 | | |
| 024 | 0.926 | 55 | 0.927 | 50 | 0.927 | 61 | 0.925 | 59 | | |
| 032 | 0.847 | 55 | 0.846 | 56 | 0.845 | 67 | 0.844 | 61 | | |
| 016 | 0.813 | 14 | 0.779 | 36 | | | | | | |

Notes: *hkl*₀ max is 999, bold values are strong lines.

^aSynthetic material.

sealed-tube X-ray source (MoK α radiation, $\lambda = 0.71073$ Å) operating at 45 kV and 35 mA. A hemisphere of intensity data was collected at room temperature in 1265 frames with ω scans (frame width 0.30° and exposure time of 10 s per frame) in the range of reciprocal space, h : -4 to 3 , k : -3 to 4 , l : -7 to 7 . A total of 329 diffraction measurements were collected. The data were corrected for Lorentz, polarization and extinction absorption calibration using the Bruker program SADABS. The diffraction data with $F_o > 3\sigma F_o$ were considered as observable points and after the data were merged, a total of 62 unique diffractions [$R(\text{int}) = 0.018$] was used for crystal-structure determination and refinement.

The space group of linzhiite was obtained by XPREP in SHELXTL 5.1 FOR NT crystal structure analytic software package. The crystal structure was solved by direct methods in $P4/mmm$ space group using SHELXLS (Sheldrick, 2008). Once the positions of Si and Fe atoms had been determined, the anisotropic displacement parameters and occupancy were refined. Finally, the crystal structure of linzhiite was refined to $R[F^2 > 2\sigma(F^2)] = 0.043$, $wR2(F^2) = 0.104$, $\Delta\rho_{\text{max}}(\text{e}\text{\AA}^{-3}) = 1.23$; $\Delta\rho_{\text{min}}(\text{e}\text{\AA}^{-3}) = -1.32$.

Linzhiite has a $P4/mmm$ structure with the cell parameters: $a = 2.696(1)$ Å, $b = 2.696(1)$ Å, $c = 5.147(6)$ Å, $Z = 1$, $V = 37.41(14)$ Å³, $D = 4.972$ g/cm³. Atom parameters are given in Table 4. The Fe–Si distance in the structure is $2.364(2)$ Å, the Fe–Fe distance is $2.696(2)$ Å and the Si–Si distance is $2.350(2)$ Å. The structure is illustrated in Fig. 3 and 4.

The crystal structure of linzhiite can be seen as a layer packing structure (Fig. 4), in which the layers are stacked along [001]. Layers consist of face-sharing FeSi_8 slightly elongated cubes; cube edges measure 2.823 Å and 2.725 Å (Si–Si distance along [001] and [100], respectively). In the stacking direction, adjacent layers are connected through Si–Si interactions (Si–Si distance 2.350 Å).

7. Discussion

The synthesis of the compounds FeSi_2 phase have two polymorphs. FeSi_2 phase is tetragonal $P4/mmm$ space-group with unit-cell $a = 2.684$ Å, $b = 2.684$ Å, $c = 5.128$ Å (Aronsson, 1960), another orthorhombic phase, $\beta\text{-FeSi}_2$ is $Cmca$ space-group with unit-cell $a = 9.863(7)$ Å, $b = 7.791(6)$ Å, $c = 7.833(6)$ Å (Dusausoy & Protas, 1971). Two corresponding naturally occurring minerals, linzhiite and luobusaite ($\beta\text{-FeSi}_2$), were found in the Luobusha chromitite. Another naturally occurring Fe–Si mineral, naquite (FeSi , IMA 2010-010), was found in the

Table 4. Atom coordinates and equivalent isotropic displacement parameters for linzhiite.

| Atom | x | y | z | $U(eq)$ |
|------|-----|-----|-----------|----------|
| Fe | 1/2 | 1/2 | 0 | 0.007(1) |
| Si | 0 | 0 | 0.2717(6) | 0.003(1) |

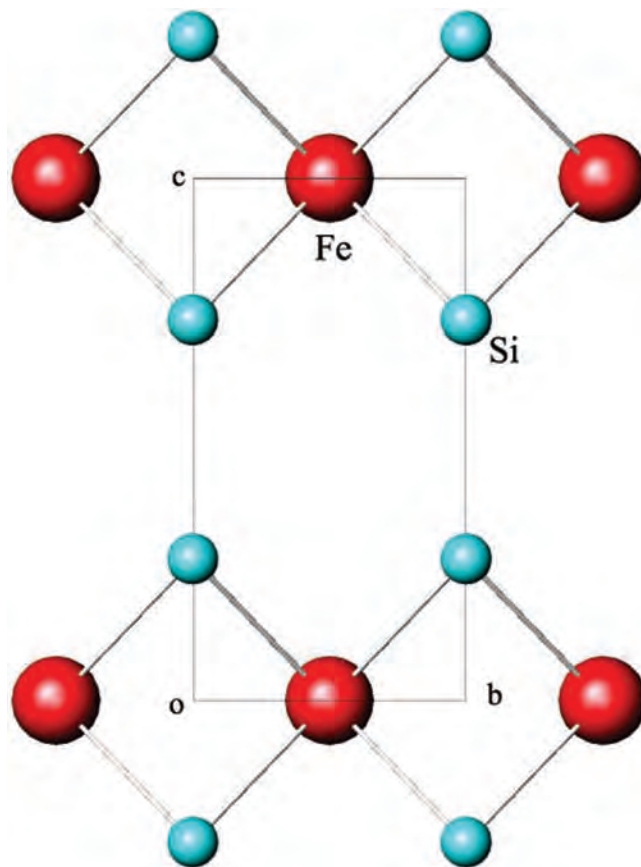


Fig. 3. The cell structure of linzhiite.

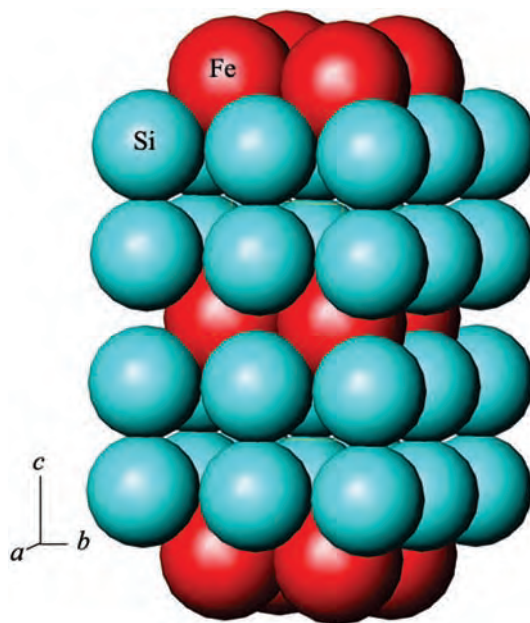


Fig. 4. The crystal structure of linzhiite.

Luobusha chromitite, as well. Other intermetallic Fe–Si minerals, approved by the IMA, are gubeiite (Fe_3Si) and xifengite (Fe_5Si_3). Their crystal parameters and composition are listed in Table 5. The characteristics of Fe–Si

Table 5. Crystal chemistry parameters for native Si–Fe alloy minerals.

| Name of minerals | Luobushaite | Linzhiite | Naquite | Gubeiite | Xifengite |
|---------------------------------------|------------------------------------|-------------------|--------------------------|--------------------|---------------------------------|
| Formula | Fe _{0.84} Si ₂ | FeSi ₂ | FeSi | Fe ₃ Si | Fe ₅ Si ₃ |
| Crystal system | Orthorhombic | Tetragonal | Cubic | Cubic | Hexagonal |
| Unit-cell parameters | | | | | |
| <i>a</i> (Å) | 9.874 (14) | 2.696 (1) | 4.452 (7) | 5.644 | 6.7416 |
| <i>b</i> (Å) | 7.784 (5) | | | | |
| <i>c</i> (Å) | 7.829 (7) | 5.147 (6) | | | 4.7079 |
| <i>V</i> (Å ³) | 601.7(8) | 37.41 (14) | 88.28 | 179.79 | 185.3 |
| <i>Z</i> | 16 | 1 | 4 | 4 | 2 |
| Space group | <i>Cmca</i> | <i>P4/mmm</i> | <i>P 2₁3</i> | <i>Fm3m</i> | <i>P6₃/mcm</i> |
| Chemical composition (electron probe) | | | | | |
| Si | 54.58 (11) | 50.00 (60) | 33.85 | 14.10 (5) | 23.70 (3) |
| Fe | 45.35 (11) | 49.09 (64) | 66.17 | 84.80 (5) | 75.50 (3) |
| Ni | | | | 0.80 (5) | 0.30 (3) |
| Mn | | 0.28 (11) | | 0.70 (5) | 0.20 (3) |
| Al | | 0.64 (28) | | | |
| Total | 99.93 | 100.01 | 100.02 | 100.4 | 99.7 |
| Data source | Li <i>et al.</i> (2007a) | This study | Li <i>et al.</i> (2007b) | ICSD53545 | ICSD42585 |

system minerals are high symmetry and small cell volume. The atoms tend to the closest packed arrangement in the crystal structure. The bonds between Fe and Si tend to be covalent, and the Fe–Si bond lengths are 2.288–2.388 Å. They are classed as ‘‘01.01.23 Suessite Group silicides’’ in the Dana Classification System.

Although linzhiite and other intermetallic compounds were collected from the podiform chromitite in an ophiolitic harzburgite, it is unlikely they were crystallized from the melt that formed the Luobusha chromitites, given the chemical incompatibilities that would require a strongly reducing environment. They are likely xenocrysts derived from mantle sources, transported up by a plume and incorporated in the ophiolite during seafloor spreading. Blocks of the mantle containing the exotic minerals were presumably picked up by later boninitic melts from which the chromitites precipitated, transported to a shallow depth and partially digested in the melt, with insoluble residues incorporated into the chromitite (Robinson *et al.*, 2004; Yang *et al.*, 2007; Robinson & Yang, 2008).

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