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The synthesis, and crystal and magnetic structure of the iron selenide BaFe$_2$Se$_3$ with possible superconductivity at $T_c = 11$ K

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Corrigendum: The synthesis, and crystal and magnetic structure of the iron selenide \( \text{BaFe}_2\text{Se}_3 \) with possible superconductivity at \( T_c = 11 \text{ K} \)

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Owing to a technical mistake, we erroneously used the wrong computer file for plotting the magnetic structure of the solution that we found for the irreducible representation (irrep) \( \tau_2 \), which was shown in figure 4(b) in the original article. Table 3, showing also the details of the magnetic structure refinements for \( \tau_2 \), and figure 4(a) were correct. The corrected figure 4 is shown in this corrigendum. The structure in (b) looks similar to that in (a), but the geometries of the Fe plaquettes forming ferromagnetic four-spin blocks are slightly different for the (a) and (b) cases of different (\( \tau_1 \) and \( \tau_2 \)) symmetry. For completeness, we explicitly list the symmetry operators, coordinates and spin directions +1 or −1 along the \( a \)-axis for all eight Fe atoms in the zeroth cell for the (a) and (b) structures.

<table>
<thead>
<tr>
<th>Symmetry operator</th>
<th>( x, y, z )</th>
<th>Spin direction for ( a, b )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( x, y, z )</td>
<td>0.494, 0.001, 0.353</td>
<td>+1, +1</td>
</tr>
<tr>
<td>( -x + 1/2, -y, z + 1/2 )</td>
<td>0.006, 0.999, 0.853</td>
<td>−1, +1</td>
</tr>
<tr>
<td>( -x, y + 1/2, -z )</td>
<td>0.506, 0.501, 0.647</td>
<td>−1, +1</td>
</tr>
<tr>
<td>( x + 1/2, -y + 1/2, -z + 1/2 )</td>
<td>0.994, 0.499, 0.147</td>
<td>−1, −1</td>
</tr>
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<td>( -x, -y, -z )</td>
<td>0.506, 0.999, 0.647</td>
<td>−1, −1</td>
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<tr>
<td>( x + 1/2, y, -z + 1/2 )</td>
<td>0.994, 0.001, 0.147</td>
<td>+1, −1</td>
</tr>
<tr>
<td>( x, -y + 1/2, z )</td>
<td>0.494, 0.499, 0.353</td>
<td>−1, +1</td>
</tr>
<tr>
<td>( -x + 1/2, y + 1/2, z + 1/2 )</td>
<td>0.006, 0.501, 0.853</td>
<td>−1, −1</td>
</tr>
</tbody>
</table>

Figure 4. Refined magnetic structure models ((a) and (b)) for Ba123. The magnetic structures for \( \tau_1 \) (a) and \( \tau_2 \) (b) are shown in the projection on the \( bc \)-plane. The black and red circles correspond to the up and down spin directions. The Se\textsubscript{2}Fe\textsubscript{1} double chains separated by a distance of approximately \( a/2 \) along the \( a \)-axis are shown with a different background color.
FAST TRACK COMMUNICATION

The synthesis, and crystal and magnetic structure of the iron selenide BaFe$_2$Se$_3$ with possible superconductivity at $T_c = 11$ K

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Abstract
We report on the synthesis of single crystals of BaFe$_2$Se$_3$ and study their crystal and magnetic structures by means of synchrotron single-crystal x-ray and neutron powder diffraction. The crystal structure has orthorhombic symmetry and consists of double chains of FeSe$_4$ edge connected tetrahedra intercalated with barium. Below 240 K, long range spin-block checkerboard antiferromagnetic order is developed. The magnetic structure is similar to one observed in A$_{0.8}$Fe$_{1.6}$Se$_2$ ($A$ = K, Rb or Cs) superconductors. The crystals exhibit a transition to the diamagnetic state with an onset transition temperature of $T_c \sim 11$ K. Though we observe FeSe as an impurity phase (<0.8% mass fraction) it is not likely that the diamagnetism is attributable to the FeSe superconductor, which has $T_c \approx 8.5$ K.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

With the discovery of alkali metal intercalated FeSe superconductor [1] with the transition temperature $T_c \approx 30$ K, a chalcogenide analog to the 122 iron pnictide superconductors [2, 3] was found. Shortly after the first reported synthesis [1] of K$_{0.8}$Fe$_2$Se$_2$ (K122), further compounds with the formula A$_x$Fe$_{2-y}$Se$_2$ intercalated with $A$ = Cs [4], Rb [5], Ti/K [6] and Ti/Rb [7] were successfully prepared. For all of these superconductors, critical temperatures around 30 K were found. All the intercalated chalcogenides, in contrast to the pnictides, can be relatively easily grown as high quality single crystals by the Bridgman method. This is extremely important, as for many studies, e.g. neutron scattering experiments, cm$^3$ size single crystals are required. For Cs$_{0.8}$Fe$_{2-y}$Se$_2$ and K$_{0.8}$Fe$_{2-y}$Se$_2$ it was found that the empirical dependence of $T_c$ on the anion height (the distance between Fe and the chalcogenide/pnictide layers), as stated for pnictides [8], is also fulfilled [4]. Thus, further increasing $T_c$ (the maximum value for pnictides is ~55 K) by applying pressure could potentially be possible. Unfortunately, a negative pressure effect with suppression of the superconductivity above 8 GPa for Cs$_{0.8}$Fe$_{2-y}$Se$_2$ was observed [9]. Further, for the A$_x$Fe$_{2-y}$Se$_2$, coexistence of superconductivity and a strong magnetism ($T_N \approx 478.5$ K) was found [10]. On the basis of neutron scattering experiments, structure models revealing iron vacancies and magnetic orderings were proposed [11–14].
In our attempt to explore ternary iron chalcogenides we gave attention to alkaline earth metal families. According to Iglesias and Steinfink [15], in the case of the barium transition metal chalcogenides Ba₄MₓX₄ (where M = 3d metal, X = Se or S), compounds with different stoichiometries x:y:z of 2:1:3, 1:1:2, 1:4:3, 1:2:2 and 1:2:3 could be synthesized. For all these compounds, nine different structure types have been found, all of them based on columns of face-sharing BaX₆ triangular prisms (tetrahedra). In BaMX₂ (Ba112) and BaM₂X₂ (Ba122), sheets are created by edge sharing of the neighboring columns, whereas both in BaM₂X₃ (Ba123) and in BaM₄X₃ (Ba143), the columns are isolated [15]. In particular, Ba123 seems to be an interesting analog to K122, as the additional charge of the alkaline earth metal is balanced by the additional chalcogenide anion in the formula. Figure 1 shows the crystal structure model of orthorhombic Ba123, emphasizing the coordination of the Fe atoms. In contrast to the case for alkali metal intercalated chalcogenides (like K122), the Fe–Se layers are here not two dimensional, but consist of double chains of FeSe₄ edge connected tetrahedra.

In this work, BaFe₂Se₃ (Ba123) single crystals were grown for the first time by the Bridgman method. In contrast to the K122 single crystals, the Ba123 ones are stoichiometric, showing no vacancies in either alkaline earth or iron sublattices. Further, the iron–selenium layer in the structure has Fe₂Se₃ stoichiometry and is created from double chains of FeSe₄ edge connected tetrahedra. Ba123 shows long range antiferromagnetic order at temperatures below 250 K and traces of superconductivity, with the onset at Tc ≈ 11 K.

2. Experimental details

Single crystals of BaFe₂Se₃ were grown from the melt using the Bridgman method. High purity (at least 99.99%, Alfa) powders of iron and selenium were mixed in the appropriate proportions and pressed into rods. For the single-crystal synthesis, a piece of Fe₂Se₃ rod was sealed in a double-wall evacuated silica ampule with a stoichiometric amount of pure barium. The ampule was annealed at 1150 °C for 24 h for homogenization. Afterwards the melt was cooled down to 750 °C with a rate of 6 °C h⁻¹ and kept at this temperature for the next 20 h. Finally the furnace was cooled down to room temperature at the rate 200 °C h⁻¹. Well-formed black crystal rods of 7 mm diameter (the inside diameter of the quartz ampule) were obtained. The crystals could be easily cleaved into plates with flat shiny surfaces. The phase purity of the crystals grown was characterized by means of powder x-ray diffraction (XRD) using a D8 Advance Bruker AXS diffractometer with Cu Kα radiation. For these measurements a fraction of the crystal was cleaved, pulverized, and loaded into the low background airtight specimen holder. All the work was performed in a He glove box to protect the powder from oxidation.

The homogeneity and elemental composition of the cleaved crystals were studied using x-ray fluorescence spectroscopy (XRF, Orbis Micro-XRF Analyzer, EDAX). Elemental distribution maps for Ba, Fe and Se were collected in vacuum, applying white x-ray irradiation produced by an Rh tube (35 kV and 500 μA). The x-ray primary beam was focused to a spot of 30 μm diameter. A primary-beam Ti filter (25 μm thickness) was applied. An area of ~0.5 cm² was scanned. Prior to the measurements, elemental calibration was carried out using as a standard a carefully weighted, homogenized mixture of Se, Fe and BaCO₃, pressed into pellets. The calibration procedure applied results in ~1.5% accuracy of the stoichiometric coefficient values determined.

Single-crystal x-ray data for BaFe₂Se₃ were collected on the SNBL beamline BM1A at the ESRF synchrotron in Grenoble (France) with a KUMA6 diffractometer equipped with the TITAN CCD detector, with λ = 0.69736(1) Å, using a combination of o- and ϕ-scans in 1° steps and a readout pixel resolution of 62 μm. The sample was mounted in a cryo-loop and cooled down to 150 K with an Oxford Cryostream 700+ N₂ blower. The distance from the crystal to the detector was calibrated using the lattice dimensions of the powder sample prepared from the crushed crystals. The intensities were integrated using CrysAlis [16]. The empirical absorption correction was made using the SADABS procedure [17]. The structure solution was performed with SHELXS and the structure refinement with SHELXL97 [18]. Powder diffraction data were collected at the MAR345 detector at the same beamline as the single-crystal data (SNBL, ESRF). The crystal-to-detector distance was calibrated with the LaB₆ NIST standard, the data were processed with Fit2D [19] and local software was used to estimate the standard deviations of the measured intensities. Neutron powder diffraction experiments (NPD) were carried out at the SINQ spallation source of the Paul Scherrer Institute (PSI, Switzerland) using the high resolution diffractometer for thermal neutrons, HRPT [20], with a neutron wavelength λ = 1.886 Å. The sample was loaded into a vanadium container with an indium seal in a He glove box. Refinements of the crystal and magnetic structures from the powder diffraction data were carried out with the FULLPROF.
program [21], with the use of its internal tables for scattering lengths and magnetic form factors. The ac magnetization measurements were performed using a quantum design PPMS magnetometer.

3. Results and discussion

Micro-XRF analysis performed on the BaFe2Se3 crystals confirmed their homogeneity. The stoichiometric coefficients determined on the basis of micro-XRF mapping were $0.99 \pm 0.02, 1.99 \pm 0.04$ and $3.00 \pm 0.05$ for Ba, Fe and Se, respectively.

Figure 2 shows three slices of the reciprocal maps normal to the $a^*$ direction, while the $hk0$ section shows much narrower profiles. This observation is consistent with the morphology of the single crystals, which are assembled from numerous blocks misaligned by a rotation, presumably around the longest $a$-axis. We have also noted a weak rod-like diffuse scattering propagating along $a^*$ that could be tentatively attributed to the stacking faults normal to the $a$ direction. For many crystals tested, the diffraction data can be successfully indexed assuming a few twin components rotated by a few degrees relative to each other. The crystal morphology, mosaicity and diffuse signal agree with a quasi-1D character of the crystal structure, that is built from covalently bound double chains of FeSe$_4$ tetrahedra weakly packed together with Ba ions.

The results of the single-crystal structural refinement of BaFe$_2$Se$_3$ are summarized in tables 1 and 2. The structural model proposed from the single-crystal experiment fitted well to the powder x-ray and neutron diffraction data (figure 2). Notably, there is one very weak additional reflection at $d \approx 5.52$ Å at $T = 300$ K seen only in the x-ray powder diffraction pattern that is not modeled with the structure of BaFe$_2$Se$_3$ ($Pnma$ space group). This peak can be tentatively assigned to FeSe$_{1.974}$ with the lattice constants $a = 3.7738, c = 5.5235$ Å [21]. Refinement of this impurity phase with the structure model from [22] gives a 0.8% mass fraction. At odds with the powder data case, a summation of the observed frames in the single-crystal experiment does not reveal an additional reflection at about 5.52 Å; such a procedure implies an averaging of the single-crystal data set over all orientations of the same crystal and is similar to the powder averaging over many crystals.

Figure 2. Three slices of the reciprocal space for the Ba123 single crystal obtained by means of synchrotron diffraction at $T = 150$ K.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\times 10^3$ Å$^2$) for 150 K. $U(^{eq})$ is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>$U(^{eq})$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ba(1)</td>
<td>1854(1)</td>
<td>2500</td>
<td>5223(1)</td>
<td>16(1)</td>
</tr>
<tr>
<td>Se(2)</td>
<td>3569(2)</td>
<td>2500</td>
<td>2308(2)</td>
<td>13(1)</td>
</tr>
<tr>
<td>Se(3)</td>
<td>6285(2)</td>
<td>2500</td>
<td>4902(2)</td>
<td>14(1)</td>
</tr>
<tr>
<td>Se(4)</td>
<td>4007(2)</td>
<td>2500</td>
<td>8158(2)</td>
<td>16(1)</td>
</tr>
<tr>
<td>Fe(5)</td>
<td>4937(2)</td>
<td>7(3)</td>
<td>3526(2)</td>
<td>13(1)</td>
</tr>
</tbody>
</table>

The results of the single-crystal structural refinement of BaFe$_2$Se$_3$ are summarized in tables 1 and 2. The structural model proposed from the single-crystal experiment fitted well to the powder x-ray and neutron diffraction data (figure 2). Notably, there is one very weak additional reflection at $d \approx 5.52$ Å at $T = 300$ K seen only in the x-ray powder diffraction pattern that is not modeled with the structure of BaFe$_2$Se$_3$ ($Pnma$ space group). This peak can be tentatively assigned to FeSe$_{1.974}$ with the lattice constants $a = 3.7738, c = 5.5235$ Å [21]. Refinement of this impurity phase with the structure model from [22] gives a 0.8% mass fraction. At odds with the powder data case, a summation of the observed frames in the single-crystal experiment does not reveal an additional reflection at about 5.52 Å; such a procedure implies an averaging of the single-crystal data set over all orientations of the same crystal and is similar to the powder averaging over many crystals.

On cooling below $T = 240$ K, a set of additional Bragg peaks is observed in the NPD patterns that can be indexed with the propagation vector $k = [1/2, 1/2, 1/2]$. The $Pnma$ space group with $k = [1/2, 1/2, 1/2]$ has two two-dimensional irreducible representations (irreps) that both enter the reducible magnetic representation for the Fe spin sitting in the general (8d) position six times. We have sorted all possible symmetry adapted solutions, with the restriction that the Fe spins have the same magnitude and are oriented either parallel or antiparallel to each other and directed along the crystal axes. There are two solutions for both $\tau_1$ and $\tau_2$ irreps that fit the experimental NPD pattern, as shown in figure 3, excellently, with only one refinable parameter—the moment value. The fit reliability

<table>
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<tr>
<th></th>
<th>$U^{11}$</th>
<th>$U^{22}$</th>
<th>$U^{33}$</th>
<th>$U^{12}$</th>
<th>$U^{13}$</th>
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<tr>
<td>Ba(1)</td>
<td>24(1)</td>
<td>6(1)</td>
<td>18(1)</td>
<td>0</td>
<td>3(1)</td>
</tr>
<tr>
<td>Se(2)</td>
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<td>4(1)</td>
<td>15(1)</td>
<td>0</td>
<td>-3(1)</td>
</tr>
<tr>
<td>Se(3)</td>
<td>23(1)</td>
<td>5(1)</td>
<td>13(1)</td>
<td>0</td>
<td>-1(1)</td>
</tr>
<tr>
<td>Se(4)</td>
<td>23(1)</td>
<td>6(1)</td>
<td>18(1)</td>
<td>0</td>
<td>5(1)</td>
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<tr>
<td>Fe(5)</td>
<td>17(1)</td>
<td>8(1)</td>
<td>12(1)</td>
<td>0(1)</td>
<td>0(1)</td>
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</table>
Table 3. Crystal and magnetic structure parameters of BaFe$_2$Se$_3$ refined from NPD at 2 K in the Pnma space group. The isotropic atomic displacement parameter $B$ is in Å$^2$, $\chi^2$ is the global chi-square values (Bragg contribution), $R_B$ is a reliability Bragg factor, given for the nuclear and magnetic phases separately, and $m$ is the magnetic moment amplitude in $\mu_B$—all are given for two magnetic models ($\tau_1$, $\tau_2$).

<table>
<thead>
<tr>
<th>$a$ (Å)</th>
<th>$b$ (Å)</th>
<th>$c$ (Å)</th>
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</thead>
<tbody>
<tr>
<td>11.890 33(14)</td>
<td>5.407 98(5)</td>
<td>9.137 52(11)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>$x$</th>
<th>$y$</th>
<th>$z$</th>
<th>$B$</th>
<th>Mult.</th>
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</thead>
<tbody>
<tr>
<td>Ba1</td>
<td>0.185 14(30)</td>
<td>1/4</td>
<td>0.522 08(51)</td>
<td>0.442(72)</td>
</tr>
<tr>
<td>Fe1</td>
<td>0.494 04(15)</td>
<td>0.001 48(38)</td>
<td>0.352 63(18)</td>
<td>0.695(25)</td>
</tr>
<tr>
<td>Se1</td>
<td>0.355 03(25)</td>
<td>1/4</td>
<td>0.232 84(32)</td>
<td>0.777(56)</td>
</tr>
<tr>
<td>Se2</td>
<td>0.630 36(21)</td>
<td>1/4</td>
<td>0.490 47(31)</td>
<td>0.434(50)</td>
</tr>
<tr>
<td>Se3</td>
<td>0.401 49(23)</td>
<td>1/4</td>
<td>0.817 31(29)</td>
<td>0.398(47)</td>
</tr>
</tbody>
</table>

| $m$ || $a$ Fe1 | $\chi^2$ | $R_B$(nucl) | $R_B$(magn) |
|-----|-----|-----|---------|---------|
| $\tau_1$ | 2.101 (13) | 3.82 | 3.04 | 4.76 |
| $\tau_2$ | 2.101 (13) | 3.82 | 3.03 | 4.84 |

$R$-factors for these two models are the same in the refinements of the powder diffraction data. All other magnetic models give significantly poorer $R$-factors (30–60%). The Fe spins have moment 2.1 $\mu_B$ and are directed along the $a$-axis, i.e. perpendicular to the Fe$_3$Se$_2$ double chains. The projection of the magnetic structure on the $bc$-plane is shown in figure 4. It is interesting that the magnetic configuration $\tau_1$ with the four spins forming a ferromagnetic block is similar to the spin-block checkerboard structure observed in A$_{0.8}$Fe$_1.6$Se$_2$ ($A = K, Rb$ or Cs) [11–14]. A description of the structure model and the magnetic and structure parameters and the details of the refinements at 2 K are summarized in table 3.

Figure 4. Refined magnetic structure models ((a) and (b)) for Ba123. The magnetic structures for $\tau_1$ (a) and $\tau_2$ (b) are shown in the projection on the $bc$-plane. The black and red circles correspond to the up and down spin directions. The Se$_2$Fe$_3$ double chains separated by a distance of approximately $a/2$ along the $a$-axis are shown with a different background color.

Figure 5 shows the temperature dependence of the magnetic moment on Fe. It is evident that the long range antiferromagnetic (AFM) order disappears quite abruptly above 240 K. However the short range AFM correlations persist up to room temperature. At 240 K the magnetic peak width starts to increase and at 250 K and higher, the peak width is about ten times larger than that at low temperature (see figure 5b). The domain size estimated from...
the AFM Bragg peak broadening amounted to >650 Å at low temperature and about 20 Å at room temperature.

Figure 6 presents the temperature dependence of the real part of the ac magnetic susceptibility for BaFe$_2$Se$_3$ (black squares) together with FeSe$_{0.98}$ (red circles).

4. Summary

Successful crystal growth of the alkaline earth intercalated iron selenide BaFe$_2$Se$_3$ (Ba123) is reported. The crystal structure is similar to that of the analog alkali metal intercalated chalcogenides, with the difference being the one-dimensional arrangement of the FeSe$_4$ edge connected tetrahedra creating double chains running along the b-axis within the Fe$_2$Se$_3$ bc-layers. AFM long range order is observed below $T_N \approx 240$ K. The AFM structure contains four-spin ferromagnetic blocks oriented antiparallel along the Fe$_2$Se$_3$ chains with Fe moments of 2.1 $\mu_B$ along the a-axis. Short range AFM order is preserved at least up to room temperature. The diamagnetic transition with an onset at about 11 K suggests the possible existence of a superconducting state.

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