

УДК 517.9

## Synthesis and Crystal Structure of the Ordered Perovskite $\text{Pb}_2\text{FeSbO}_6$

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Received 10.11.2012, received in revised form 10.12.2012, accepted 20.01.2013

*This paper describes the synthesis of perovskite-like compound  $\text{Pb}_2\text{FeSbO}_6$  which undergoes a phase transition between 210–220 K. According to X-ray studies of the powder sample the structure of the initial cubic and the low-temperature tetragonal phases of  $\text{Pb}_2\text{FeSbO}_6$  were determined. The results obtained have been discussed using the group-theoretical analysis.*

*Keywords: phase transition, irreducible representations, X-ray experiment, perovskites, crystal, ceramics.*

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## Introduction

Many oxides of  $\text{A}_2\text{BB}'\text{O}_6$  type (where A — La, Ca, Sr, Ba, Pb; B, B' — Ni, Ru, Sb, Mo, Cd, Ta, Ho, La, Te, Er, Cu, W, Fe, Tm, Bi, Pr, Tb, Yb, Li) crystallize in an ordered perovskite

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structure (elpasolite), and may undergo a phase transition (PT) of different nature [1–3].  $\text{AX}_3$  layers form elpasolite-like crystals  $\text{A}_2\text{BB}'\text{X}_6$  (where  $\text{X} = \text{Cl}, \text{F}, \text{O}$ ). All the octahedral voids of this package are filled by cations B and B', alternating along the axes of the cube. Octahedra  $\text{BX}_6$  and  $\text{B}'\text{X}_6$  have different sizes due to the shift of the X anion towards B' high-valency cation. The only arbitrary parameter of the elpasolite cubic structure is one of the coordinates of the ion X. Elpasolite cubic phase has FCC Bravais lattice, with the parameter of the unit cell twice of the perovskite's one.

The ratio of the ionic radii and charges of cations B and B' affect the nature of the ordering, and thus the structural characteristics and physical properties. These crystals belong to the space group  $G_0 = O_h^5 (Fm\bar{3}m)$ , even in cases when the ordering of B and B' ion is only partial. Ordered compounds, which include oxygen elpasolites undergo «sharp» (nondiffused) transformations, the sequence of which depends strongly on the type of cations [1–3].

The recently synthesized new compound  $\text{Pb}_2\text{FeSbO}_6$  is related to the family described above, and does not crystallize in the perovskite structure under normal conditions. Preliminary studies of  $\text{Pb}_2\text{FeSbO}_6$  ceramics revealed the presence of a diffuse dielectric constant maximum at 210–220 K, its temperature being independent on frequency of the measuring field. These results indicate the presence of ferroelectric or antiferroelectric phase transition in  $\text{Pb}_2\text{FeSbO}_6$ . This paper reports the synthesis of the perovskite modification of  $\text{Pb}_2\text{FeSbO}_6$  under high pressure, and changes in the structure of  $\text{Pb}_2\text{FeSbO}_6$  in the temperature range of 130–300 K, which includes the PT region.

## 1. Synthesis of the $\text{Pb}_2\text{FeSbO}_6$ compound

The starting reagents for the synthesis of  $\text{Pb}_2\text{FeSbO}_6$  compound were  $\text{PbO}$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{Sb}_2\text{O}_5$  oxides (A.C.S. grade). The synthesis was performed in two stages. At the first stage by the means of pressing tablets were formed from the mixture of ball-milled powders of oxides corresponding to the stoichiometric composition of the target compound. After that, the tablets were subjected to heat treatment at 1020–1030 K for 4 hours in a closed corundum crucible filled by a mixture of lead and zirconium oxides. After the first stage of the synthesis, as shown by X-ray analysis, the resulting compound has the pyrochlore structure.

Product of the first stage of synthesis was ground, and then ball-milled. The tablets for the second stage of the synthesis were pressed from the resulting charge. The second stage of the synthesis was performed at the pressure of 6 GPa and the temperature of 1400–1500 K for 5 min. After the synthesis, the system was rapidly cooled to the room temperature under pressure. Then the high-pressure apparatus was unloaded. The product of the second high-pressure synthesis stage was dense ceramic of the perovskite phase of  $\text{Pb}_2\text{FeSbO}_6$  without any visible content of parasitic phases.

Annealing step method has shown that the resulting perovskite phase in the form of a monolithic tablets is stable when heated in air up to the temperature of 900–1000 K. The limit of the thermal stability of the perovskite phase in the powdered form is reduced to about 800–900 K. For removal of stresses the powder was annealed at 700 K for 2 hours before the X-ray diffraction studies.

## 2. The results of X-ray diffraction experiments

X-ray diffraction patterns of a powdered  $\text{Pb}_2\text{FeSbO}_6$  samples were obtained using TTK450 Anton Paar temperature camera, mounted on an X-ray D8-ADVANCE diffractometer (Cu  $\text{K}_\alpha$ -radiation,  $\theta$ - $2\theta$  scanning linear detector VANTEC were used). Liquid nitrogen was used as a refrigerant.

To improve the data quality variable counting time (VCT) and variable step scanning (VSS) methods were used in the experiment [4–6]. The advantage of these methods as compared to the conventional one is that they equalize the weights of strong low-angled reflections and those weak ones at high angles, whereas in conventional X-ray experiment weights are non-equivalent, and the structure information contained in the X-ray diffraction pattern at high angles  $2\theta$  is lost. In addition, the application of VCT and VSS methods significantly reduces the time of the experiment [7], without changing the quality of data.

Using the software, XRD Wizard [7] experimental X-ray diffraction patterns of the  $\text{Pb}_2\text{FeSbO}_6$  sample were divided into two intervals of  $2\theta$ : 1)  $2\theta$  from  $5^\circ$  to  $60^\circ$ , in the increments of  $0.016^\circ$  and exposure at each point for 4 seconds; and 2)  $2\theta$  from  $60^\circ$  to  $145^\circ$ , in the increments of  $0.032^\circ$  and exposure for 16 seconds at each point. Structure refinement of the  $\text{Pb}_2\text{FeSbO}_6$  crystal was carried out using software package [8].

Reflections positions on X-ray diffraction pattern of  $\text{Pb}_2\text{FeSbO}_6$  (Fig. 1) obtained at room temperature of  $T = 300$  K indicated FCC-cell of the  $Fm\bar{3}m$  space group.

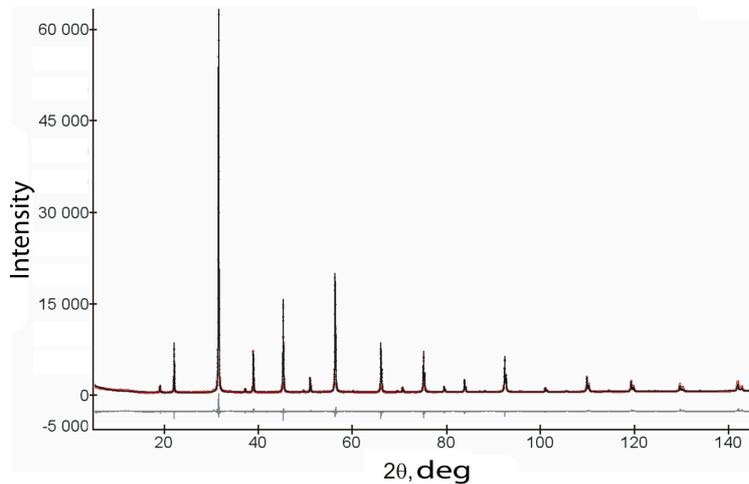


Fig. 1. X-ray diffraction pattern of the cubic  $Fm\bar{3}m$  phase  $\text{Pb}_2\text{FeSbO}_6$  at  $T = 300$  K. Red dots — experimental intensity, solid black line — calculated intensity, the gray line shows the difference between them

In the cubic phase of the crystal of the ordered elpasolite  $\text{Pb}_2\text{FeSbO}_6$  all atoms except oxygen are in special positions [3,9]. Thus from the structural parameters one coordinate of the oxygen atom, isotropic thermal parameters for all atoms, and occupancy positions for Fe and Sb were refined.

As a result of refinement it was found that the  $4a$  position with  $(0, 0, 0)$  coordinates is occupied by  $0.831(9)$  Fe ions and  $0.169(9)$  Sb ions, and the  $4b$  position  $(1/2, 1/2, 1/2)$  is occupied by  $0.848(8)$  Sb ions and  $0.153(8)$  Fe ions. That is, the total occupation for the two positions of the iron ions are  $0.831 + 0.153 = 0.984$ , and Sb ions  $0.848 + 0.169 = 1.017$ . In this case, the compound formula can be written as  $\text{Pb}_2\text{Fe}_{0.984}\text{Sb}_{1.017}\text{O}_6$ . As the  $4a$  (Fe) and  $4b$  (Sb) sites population are equal to 1 within two standard deviations, the refinement was carried out with the restriction in the linear constraints form on the occupation of these positions, so that as a result we get the formula  $\text{Pb}_2\text{FeSbO}_6$ . At the end, the distribution of ions at the  $4a$  and  $4b$  positions in the structure of the cubic phase were defined in the  $\text{Pb}_2(\text{Fe}_{0.869(4)}\text{Sb}_{0.131(4)}) (\text{Sb}_{0.869(4)}\text{Fe}_{0.131(4)})\text{O}_6$  formula. The results of the structure refinement for the  $\text{Pb}_2\text{FeSbO}_6$  cubic phase are given in Tab. 1 and 2. Table 3 presents the basic bond lengths. Fig. 7 a, shows the cubic  $\text{Pb}_2\text{FeSbO}_6$  phase projection in the plane perpendicular to the axis of the fourth order. Refined profile

parameters of the cubic phase were further used without additional changes and refinements for the solving of low-symmetry phase structure.

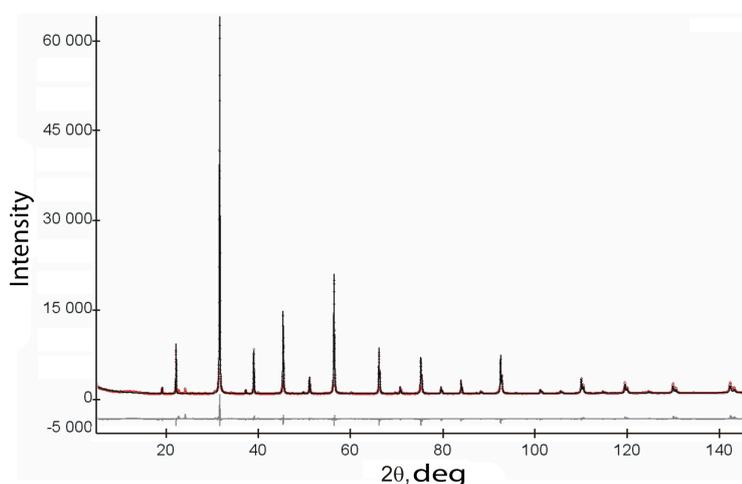


Fig. 2. X-ray diffraction pattern of the tetragonal  $I4/m$  phase  $\text{Pb}_2\text{FeSbO}_6$  at  $T = 133$  K. Red dots — experimental intensity, solid black line — calculated intensity, the gray line shows the difference between them

On the X-ray diffraction pattern of the low-temperature  $\text{Pb}_2\text{FeSbO}_6$  phase (Fig. 2), obtained at  $T = 133$  K, no superstructure reflections were observed. At low temperature, only subtle peaks of ice formed during the sample cooling could be seen.

To determine the point symmetry of the crystal low-temperature phase additional experiments in the temperature range from 143 K to 293 K with a step on a temperature of 10 K were carried out. Splitting of reflexes, by which the point of symmetry change can be determined, was not observed in X-ray diffraction patterns (Fig. 3). However, some information was obtained from the

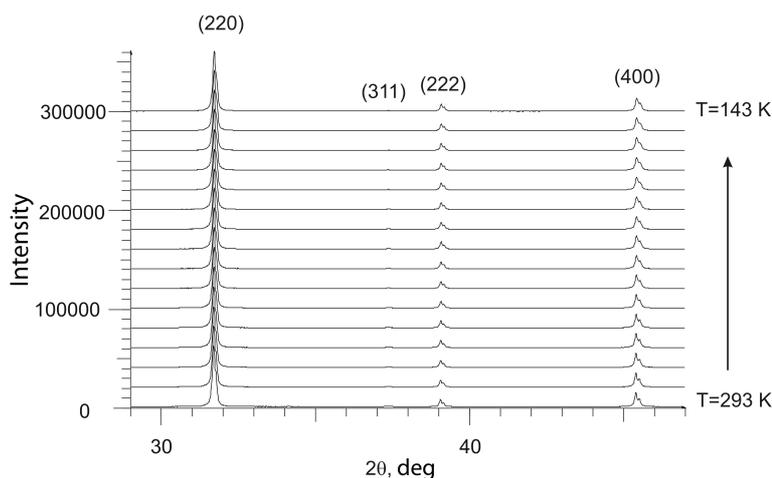


Fig. 3. Temperature evolution of the X-ray diffraction pattern of  $\text{Pb}_2\text{FeSbO}_6$ . Basic reflections of the prototype perovskite structure are shown

analysis of the full-width at half-maximum (FWHM) temperature dependence of several major

reflections (Figs. 4–6). It can be easily seen from the figures 4–6 that in the PT region reflexes broaden: (220) by 2.3% and (400) by 10.4%, while the FWHM of reflection (111) remains unchanged within the measurement error. Using the homology method [10], this broadening can be explained by the splitting of reflections, which most likely correspond to the tetragonal distortion of the face-centered cubic unit cell (Tab. 4).

Table 1. The main crystallographic data and experimental parameters for  $\text{Pb}_2\text{FeSbO}_6$  at 300 K and 133 K

Experiment temperature	300 K	133 K
Space group	$Fm\bar{3}m$	$I4/m$
$A$ , Å	7.98361(4)	5.64102(4)
$C$ , Å		7.9704(1)
$V$ , Å <sup>3</sup>	508.859(8)	253.627(5)
$Z$	4	2
$2\theta$ -range, °	5–145	5–145
Reflections number	41	132
Refinement parameters number	24	18
$R_{wp}$ , %	6.754	8.140
$R_{exp}$ , %	3.410	3.386
$R_p$ , %	5.054	5.930
$R_B$ , %	3.42	3.97
GOF ( $\chi$ )	1.981	2.404

Table 2. Atomic coordinates, occupancy  $p$ , and isotropic thermal  $B_{iso}$  parameters for  $\text{Pb}_2\text{FeSbO}_6$  at 300 K and 133 K

$T = 300$ K					
	$X$	$y$	$z$	$p$	$B_{iso}$ , Å <sup>2</sup>
Pb	1/4	1/4	1/4	1	2.2(4)
Fe <sub>1</sub>	0	0	0	0.869(4)	0.7(4)
Sb <sub>1</sub>	0	0	0	0.131(4)	0.7(4)
Fe <sub>2</sub>	1/2	1/2	1/2	0.131(4)	0.7(4)
Sb <sub>2</sub>	1/2	1/2	1/2	0.869(4)	0.7(4)
O	0.2472(9)	0	0	1	1.1(1)
$T = 133$ K					
	$X$	$y$	$z$	$p$	$B_{iso}$ , Å <sup>2</sup>
Pb	0	1/2	1/4	1	1.43(2)
Fe <sub>1</sub>	0	0	0	0.867(7)	0.86(3)
Sb <sub>1</sub>	0	0	0	0.133(7)	0.86(3)
Fe <sub>2</sub>	0	0	1/2	0.133(7)	0.86(3)
Sb <sub>2</sub>	0	0	1/2	0.867(7)	0.86(3)
O <sub>1</sub>	0.775(6)	0.733(8)	0	1	1.0(2)
O <sub>2</sub>	0	0	0.244(9)	1	1.0(2)

Further, the space group choice of low-temperature tetragonal phase was carried out using the results of Ref. [11] on the group-theoretical analysis of the structural phase transitions in

crystals with space group  $Fm\bar{3}m$  and ISODISTORT program complex [12].

Table 3. Characteristic bond lengths for  $\text{Pb}_2\text{FeSbO}_6$  at 300 K and 133 K

$T = 300 \text{ K}$		$T = 133 \text{ K}$	
bond	$d, \text{ \AA}$	bond	$d, \text{ \AA}$
$\text{Fe}_1 (\text{Sb}_1) - \text{O}$	1.973(7)	$\text{Fe}_1 (\text{Sb}_1) - \text{O}_1$	1.97(3)
		$\text{Fe}_1 (\text{Sb}_1) - \text{O}_2$	1.95(7)
$\text{Fe}_2 (\text{Sb}_2) - \text{O}$	2.019(7)	$\text{Fe}_2 (\text{Sb}_2) - \text{O}_1$	2.03(4)
		$\text{Fe}_2 (\text{Sb}_2) - \text{O}_2$	2.04(7)
$\text{Pb} - \text{O}$	2.823(5)	$\text{Pb} - \text{O}_1$	2.70(3)
		$\text{Pb} - \text{O}_2$	2.821(1)

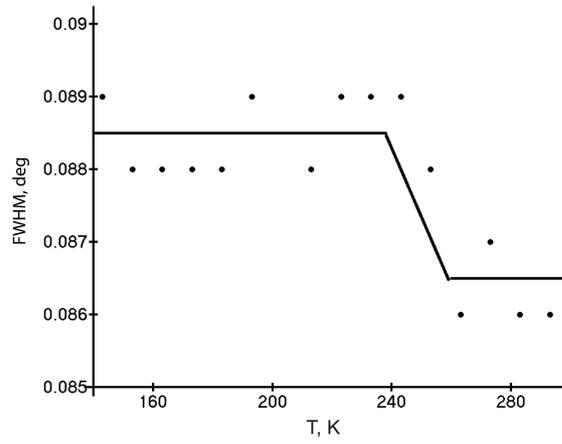


Fig. 4. Temperature dependence of the (220) reflection FWHM

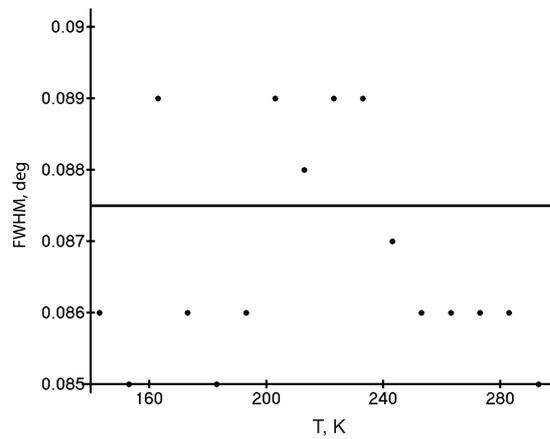


Fig. 5. Temperature dependence of the (222) reflection FWHM

Following [11, 12], all the variants of tetragonal space groups, in which the PT from  $Fm\bar{3}m$  without changing the volume of the unit cell is possible were obtained, as the superstructure

reflections in the low-temperature phase were not detected. There were six variants:  $I4/mmm$ ,  $I\bar{4}2m$ ,  $I4mm$ ,  $I4/m$ ,  $I\bar{4}$ , and  $I4$ . All cells have a basis of  $a_{tetr} = (a_{cub} + b_{cub})/2$ ;  $b_{tetr} = (a_{cub} - b_{cub})/2$ ;  $c_{tetr} = c_{cub}$ , where  $a_{cub}$ ,  $b_{cub}$ ,  $c_{cub}$  — the face-centered cubic cell basis,  $a_{tetr}$ ,  $b_{tetr}$ ,  $c_{tetr}$  — the tetragonal unit cell basis.

Since the experimental data that would help in the selection of the tetragonal phase space group were absent, the following considerations were taken into account.

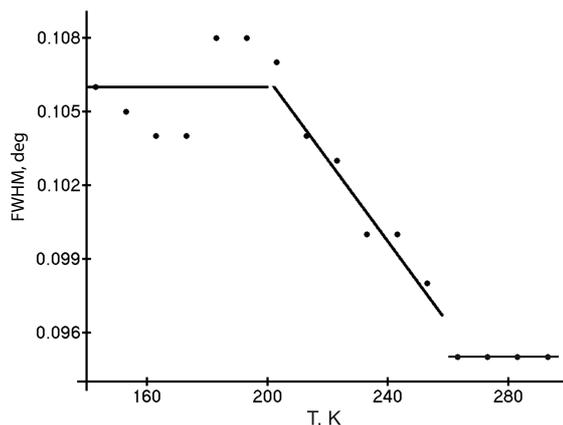


Fig. 6. Temperature dependence of the (400) reflection FWHM

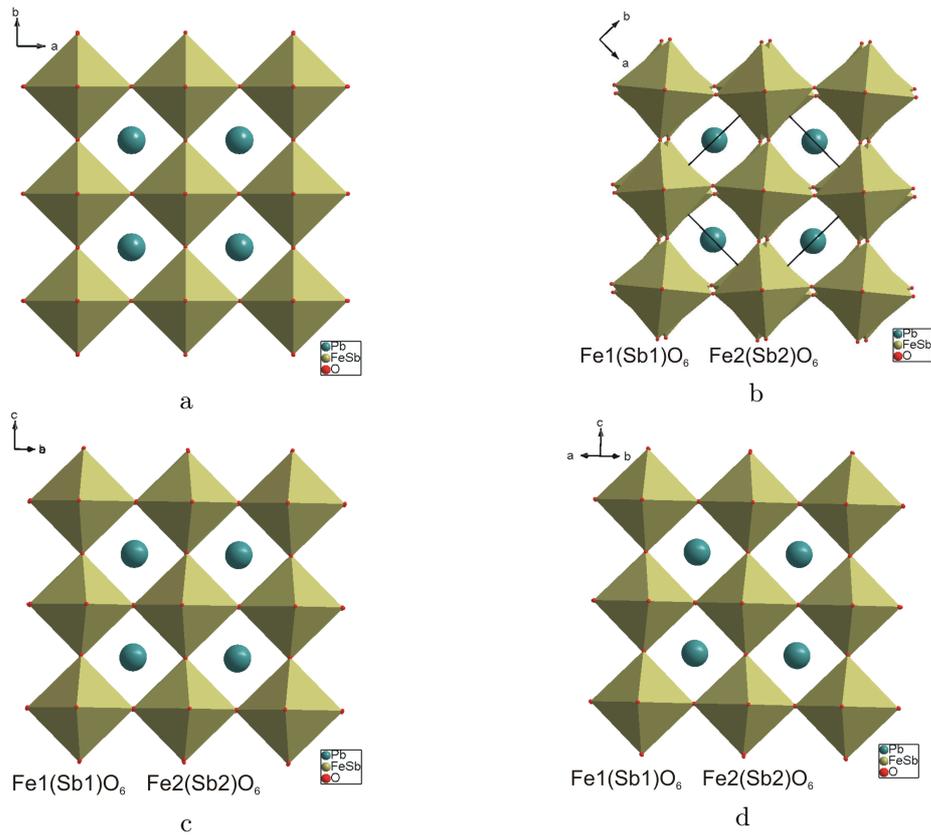
Refinement of the structure of tetragonal  $\text{Pb}_2\text{FeSbO}_6$  phase in all of the groups gave identical values within the standard deviations of the uncertainty factors. However, the number of structural parameters refined in the polar groups is significantly larger than in centrosymmetric ones. In addition, numerous studies (see, e.g. [2, 3, 9]) over an ordered perovskite (elpasolite) indicate the phases in which the critical distortions are rotations of octahedral groups, leading to structures with the presence of the center of symmetry. For these reasons, subsequently polar groups  $I4mm$  and  $I4$  were not considered. Furthermore, the transition to the space group  $I4/mmm$  is associated with the emergence of a critical order parameter (OP)  $(a, 0)$ , which transforms to  $\Gamma_3^+$  of  $Fm\bar{3}m$  representation leading to the compression/expansion of the octahedron  $\text{Fe}(\text{Sb})\text{O}_6$ . Hereafter in the article the notation of irreducible representations (IR) groups are given according to the handbook [13]. Transition to  $I\bar{4}2m$  is associated with the critical  $(a, 0, 0)$  parameter  $\Gamma_5^-$  representation, which leads to the  $\text{Fe}(\text{Sb})\text{O}_6$  polyhedron deformation. Transition to  $I\bar{4}$  is associated with the appearance of two critical  $(a, 0, 0)$  OP  $\Gamma_5^-$  representation and  $(b, 0, 0)$  OP  $\Gamma_4^+$  representations, which lead to the rotation and distortion of the  $\text{Fe}(\text{Sb})\text{O}_6$  polyhedron. More interesting is the transition to the group  $I4/m$ , in which there is a critical  $(a, 0, 0)$  OP  $\Gamma_4^+$  representation, resulting in a pure  $\varphi$ -type rotation of the  $\text{Fe}(\text{Sb})\text{O}_6$  polyhedron. Rotations are designated according to [14].

Given the considerations above, the further consideration of the structure of the tetragonal phase was carried out in the  $I4/m$  space group. Coordinates of the two independent oxygen atoms, thermal parameters of all atoms and population of the sites occupied by Fe and Sb atoms were refined from the structural parameters. The sites populations were refined with the restrictions imposed earlier, so that the  $\text{Pb}_2\text{FeSbO}_6$  formula was true. The result was the following ions distribution over the B- positions:  $\text{Pb}_2(\text{Fe}_{0.867(6)}\text{Sb}_{0.133(6)})(\text{Sb}_{0.867(6)}\text{Fe}_{0.133(6)})\text{O}_6$ . Thus, the distribution of Fe and Sb over occupied positions in the tetragonal phase has not changed in comparison to the cubic phase. That is the charge ordering has not happened. The coordinates, thermal parameters, the atoms positions populations and the bond lengths of the tetragonal  $\text{Pb}_2\text{FeSbO}_6$  phase are given in the Tab. 2 and 3. The structure projection of the tetragonal phase in the plane perpendicular to the cubic cell axis is presented in Fig. 7 b, c and d.

Table 4. Splitting of the main reflections on X-ray diffraction pattern during deformation of FCC-cell according to Mikheyev [10]

Distortion type №	Syngony	Split reflections number					
		(111)	(200)	(220)	(311)	(222)	(400)
1	<b>Tetragonal</b>	1	2	2	2	1	2
2	Trigonal	2	1	2	3	2	1
3	Rhomboid <sup>1</sup>	2	2	3	4	2	2
4	Rhomboid <sup>2</sup>	1	3	3	3	1	3
5	Monoclinic <sup>3</sup>	2	3	4	6	2	3
6	Monoclinic <sup>4</sup>	3	2	4	7	3	2
7	Triclinic	4	3	6	12	4	3

Notes:

<sup>1</sup>  $a_{orth} \parallel (a_{cub} + b_{cub})/2$ ,  $b_{orth} \parallel (a_{cub} - b_{cub})/2$ ,  $c_{orth} \parallel c_{cub}$ <sup>2</sup>  $a_{orth} \parallel a_{cub}$ ,  $b_{orth} \parallel b_{cub}$ ,  $c_{orth} \parallel c_{cub}$ <sup>3</sup>  $b_{mon} \parallel a_{cub}$ , ( $b_{mon}$  – monoclinic axis)<sup>4</sup>  $b_{mon} \parallel (a_{cub} + b_{cub})/2$ , ( $b_{mon}$  – monoclinic axis)Fig. 7. The  $\text{Pb}_2\text{FeSbO}_6$  structure: a – projection of cubic phase structure  $Fm\bar{3}m$  along a fourfold axis, b, c, d – projection of tetragonal phase structure  $I4/m$ : b – along the fourfold axis (the  $c_{tetr}$  axis), c – along  $a_{cub}$  of the initial phase (the axis of  $a_{tetr} - b_{tetr}$ ), d – along the initial phase  $b_{cub}$  (the axis of  $a_{tetr} + b_{tetr}$ ). Distortion symbol is  $(0, 0, \varphi)$

### 3. Discussion

The results are discussed using the results of the work [11] on the group-theoretical analysis of the structural phase transitions in crystals with the  $Fm\bar{3}m$  space group and ISODISTORT [12] and ISOTROPY [15] program complexes. From Tab. 2,3 and figure 7 it is clear that the critical displacement is the displacement of the oxygen atoms, which together can be seen as  $\varphi$ -type octahedral groups rotations. Such displacements are associated with the critical OP  $\Gamma_4^+$  representation.

Here we note that the critical IR and OP determine the symmetry of the distorted phase. But in addition to the critical distortions of initial phase structure in the distorted (dissymmetric) phase displacement of atoms are compatible with the symmetry of this phase and are defined by non-critical (secondary) OP and IR. The entire set of OP, both critical and non-critical, arising during PT, forms a complete condensate of OP [16].

Works [11, 12] show that together with the critical distortions of  $\varphi$ -types there should be non-critical distortion of the octahedral groups associated with  $\Gamma_3^+$  IR of  $Fm\bar{3}m$  space group. After decomposition of the atomic displacements in the tetragonal phase of the cubic phase basis functions of  $Fm\bar{3}m$  IR we got the displacements of atoms in the tetragonal phase with respect to their positions in the cubic one. So, the critical displacement of the O atoms, associated with a  $\Gamma_4^+$  critical representation, is 0.17 Å, while an offset of O atoms due to uncritical  $\Gamma_3^+$  representation is 0.02 Å, that is almost an order in magnitude smaller. So, obviously just  $\Gamma_4^+$  makes the greatest contribution to the displacement of oxygen atoms. All these displacements can be represented as  $\varphi$ -type rotations [3,9,14].

### Conclusion

Using the synthesis under high pressure, we managed to obtain the ordered  $\text{Pb}_2\text{FeSbO}_6$  perovskite phase, which remains stable when heated in air up to temperature of 900–1000 K.

X-ray powder diffraction techniques, involving the complete symmetry analysis of condensate of order parameters defined structural changes in the ordered  $\text{Pb}_2\text{FeSbO}_6$  perovskite. Schematically, the change of the spatial symmetry of the  $\text{Pb}_2\text{FeSbO}_6$  crystal at the phase transition is as follows:  $Fm\bar{3}m \xrightarrow[\varphi,0,0]{\Gamma_4^+} I4/m$ . The main critical changes at the phase transition are the shifts of the oxygen atoms, which together can be represented as  $\varphi$ -type rotations of octahedral groups. Thermodynamic analysis carried out the basis of Ref. [15], shows that such a change of the structure can be realized at the second order phase transition.

*This work was supported by RFBR (12-02-90019 Bel\_a), the President of the Russian Federation grant for Support of Leading Scientific Schools (NS-4828.2012.2) and the Belarusian Republican Fundamental Research Foundation (T12R-077 grant).*

### References

- [1] Ju.N.Venevtsev, E.D.Politova, S.A.Ivanov, Ferro- and Antiferroelectrics of Barium Titanate Family, Moscow, Chemistry, 1985 (in Russian).
- [2] K.S.Aleksandrov, B.V.Beznosikov, Perovskite-like crystals, Novosibirsk, Nauka, 1997 (in Russian).
- [3] K.S.Aleksandrov, B.V.Beznosikov, Perovskites: present and future. Variety of parent phases, phase transitions, possibilities of the synthesis of new compounds Novosibirsk, SB RAS, 2004 (in Russian).

- [4] I.C.Madsen, R.J.Hill, Variable step-counting times for Rietveld analysis or getting the most out of your experiment time, *Adv. X-ray Anal.*, **35**(1992), 39–47.
- [5] I.C.Madsen, R.J.Hill, Collection and analysis of powder diffraction data with near-constant counting statistics, *J. Appl. Cryst.*, **27**(1994), 385–392.
- [6] W.I.F.David, «Accuracy in Powder Diffraction: Optimization of data collection strategies» – Abstract P2.6, *NIST Special Publication* no. 846, 210.
- [7] Diffrac-Plus Basic XRD Wizard. 2002-2007 Bruker AXS GmbH, Karlsruhe, Germany.
- [8] Bruker AXS: TOPAS V4: General profile and structure analysis software for powder diffraction data. User's Manual, Bruker AXS, Karlsruhe, Germany, 2008.
- [9] S.V.Misjul, M.S.Molokeev, L.V.Osokina, I.N.Saphonov, Structural Changes during Phase Transitions, Critical and Noncritical Order Parameters in the Elpasolite  $\text{Cs}_2\text{RbDyF}_6$ , *Journal of Siberian Federal University. Mathematics & Physics*, **5**(2012), no. 4, 566–575.
- [10] V.I.Mikheev, X-ray Determinant of Minerals, Geologiya i Okhrana Nedr, Moscow, 1957 (in Russian).
- [11] K.S.Aleksandrov, S.V.Misul, E.E.Baturinets, Symmetrical Analysis of Structural Phase Transitions in Crystals with the  $O_h^5$  Space Group, *Ferroelectrics*, 2007, 60–68.
- [12] B.J.Campbell, H.T.Stokes, D.E.Tanner, D.M.Hatch, ISODISPLACE: a web-based tool for exploring structural distortions, *J. Applied Crystallography*, **39**(2006), 607.
- [13] O.V.Kovalev, Irreducible and Induced Representations and Co-Representations of Federov's Groups, Moscow, Nauka, 1986 (in Russian).
- [14] K.S.Aleksandrov, S.V.Misjul, Phase Transitions Associated with Rotational Distortions in the Crystal Structure of Perovskite, *Kristallografiya*, **26**(1981), no.5, 1074–1085 (in Russian).
- [15] H.T.Stokes, D.M.Hatch, B.J.Campbell, ISOTROPY, stokes.byu.edu/isotropy.html, 2007.
- [16] V.P.Sakhnenko, V.M.Talanov, G.M.Chechin, Group-Theoretic Analysis of the Complete Order Parameters Condensate that Occurs at Structural Phase Transitions, *Fizika Metallov i Metallovedeniya*, **62**(1986), no. 5, 847–856 (in Russian).

## Синтез и кристаллическая структура упорядоченного перовскита $\text{Pb}_2\text{FeSbO}_6$

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В статье описывается синтез перовскитоподобного кристалла  $\text{Pb}_2\text{FeSbO}_6$ , претерпевающего фазовый переход в области 210–220 К. По данным рентгеновского эксперимента, от порошкового образца определены структуры исходной кубической и низкотемпературной тетрагональной фаз кристалла. Обсуждение результатов проводится с привлечением симметричного анализа.

Ключевые слова: фазовый переход, неприводимые представления, рентгеновский эксперимент, перовскиты, кристалл, керамика.