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ANALYSIS OF THE CRYSTALLINE STRUCTURE OF POLYMORPHIC MODIFICATIONS OF COMPOUND $Ba_6Ta_2O_{11}$

The object of research is the crystal structure of polymorphic modifications of the $Ba_6Ta_2O_{11}$ compound. This compound has low dielectric losses in the microwave (microwave) range and can be used in microwave technology. The compound crystallizes in the structural type of cryolite with cubic lattice parameters $a=8.69$ Å. At the same time, $Ba_6Ta_2O_{11}$ has inherent polymorphism. The paper proposes models of crystal structures for the α - and β -phases of the compound for the spectra under the numbers 00-049-0899, and 00-049-0903 in the database of powder diffraction patterns PDF-2 for 2004. The compound has a lattice that does not belong to the tetragonal system, as suggested earlier, but to the orthorhombic one. For the α -phase, a structural model is proposed: orthorhombic system with lattice periods $a=6.218$ Å; $b=8.509$ Å; $c=6.227$ Å. The space group $P2_12_12_1$ is possible (19). Odds factor $R=8.54707$ %. For the β -phase: the orthorhombic system. The space group of symmetry $Fmmm$ (69) with lattice period is possible $a=8.668$ (7) Å; $b=8.677$ (8) Å; $c=8.685$ (7) Å. Odds factor $R=7.03646$ %. Let's assume that the phase transitions are associated not only with a change in the lattice symmetry (the appearance of the second crystal symmetry elements), but also with structural disordering. The regular systems of points of the α - and β -phases of the compound are not completely filled, which introduces defects into the crystal lattice. Thus, the structure of the α -phase of the compound is completely occupied by the positions of the B2, B5, B6, B7, Ta2, Ta3, O3, O10 atoms (it has the correct system of points 4a). The structure of the β -phase has regular systems of points 4a, 4b, 8f, 32p, completely filled with atoms. This leads to distortion of the crystal structure of the test compound. The structure of the β -phase additionally has elements of symmetry of the mirror reflection plane, perpendicular to the x , y , z axes. They have elements of symmetry: the axis of symmetry of the 2nd order, parallel to the axes x , y , z . Also, helical symmetry axes of the 2nd order (inherent in both structures), parallel to the x , y , z axes, and grazing reflection planes perpendicular to the x , y , z axes, with n -slip along the diagonal. The conducted studies of the crystal structure of the compound allow to study in more detail its physical properties, in particular, as a promising dielectric in the microwave range.

Keywords: X-ray diffraction analysis, crystal structure, Rietveld method, $Ba_6Ta_2O_{11}$ compound, polymorphic modifications.

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1. Introduction

Microwave dielectrics are characterized by low dielectric losses in the microwave range and are widely used in microwave technology. Barium tantalate, in particular $Ba_3TaO_{5.5}$ [1], is a representative of these materials. This compound is characterized by polymorphism [2]. Tantalate was obtained by calcining a mixture of barium carbonate and tantalum pentoxide at a temperature of 1200 °C. According to [3], it crystallizes in the structural type of cryolite with cubic lattice parameters $a\approx 8.69$ Å. In [2], several polymorphic modifications of this compound were found and indexed. Therefore, the study of the crystal structure of this compound remains relevant.

2. The object of research and its technological audit

The object of research is the crystal structure of polymorphic modifications of the $Ba_6Ta_2O_{11}$ compound.

$Ba_6Ta_2O_{11}$ was obtained by calcining mixtures of stoichiometric amounts of barium carbonate and tantalum pentoxide in the temperature range 600–1500 °C in air in corundum and platinum crucibles [2].

One of the most problematic areas is the presence of a large number of polymorphic modifications of this compound [2]. The PDF-2 database for 2004 contains three diffraction spectra obtained for the $Ba_6Ta_2O_{11}$ compound, two of which are indexed, the third is a spectrum of low quality.

3. The aim and objectives of research

The aim of this research is to propose structural models for the diffraction spectra of the $Ba_6Ta_2O_{11}$ compound under numbers 00-049-0899, and 00-049-0903 in the PDF-2 database for 2004. To achieve this aim, it is necessary to solve the following objectives:

1. Determine the periods of the lattice and syngony in which polymorphic modifications of the compound under study crystallize.

2. Select a space group of symmetry and propose a structural model for these compound spectra.

3. Refine the microstructure parameters for the selected model by the Rietveld method.

4. Research of existing solutions of the problem

The PDF-2 database for 2004 contains three diffraction spectra of the $Ba_6Ta_2O_{11}$ compound (Table 1).

Table 1
Information about the crystal structure of $Ba_6Ta_2O_{11}$

| No. | Compound | Syngony, SSG | Lattice periods, Å° | Card number on the PDF-2 database | Diffraction spectrum quality |
|-----|------------------|---------------|--|-----------------------------------|------------------------------|
| 1 | $Ba_6Ta_2O_{11}$ | Tetragonal, I | $a=6.2310$ $b=6.2310$ $c=8.5160$ | 00-049-0899 | Indexed [2] |
| 2 | $Ba_6Ta_2O_{11}$ | Tetragonal, I | $a=6.1760$ $b=6.1760$ $c=8.5930$ | 00-049-0902 | Low precision spectrum [2] |
| 3 | $Ba_6Ta_2O_{11}$ | Cubic, F | $a=8.6740$ $b=8.6740$ $c=8.6740$ | 00-049-0903 | Indexed [2] |

Note: SSG – space symmetry group

A fragment of the phase diagram of the BaO-MgO-Ta₂O₅ system given in [4] is shown in Fig. 1, where it is indicated that the compound is a metastable phase and can be used as a microwave dielectric [1, 4].

According to [4], the dielectric properties (dielectric figure of merit) is closely related to their crystal structure. There is also information that this compound is a metastable phase [4] and crystallizes in the $[NH_4]_3FeF_6$ [3] structural type [3].

In [5] it is indicated that the compound $Ba_6Ta_2O_{11}$ was not found in the region from $Ba_5Ta_4O_{15}$ and BaO as a stable phase.

As shown in [6], the compound under study melts congruently at a temperature of 2140 °C. The state diagram of the BaO-Ta₂O₅ system according to [6] is shown in Fig. 2.

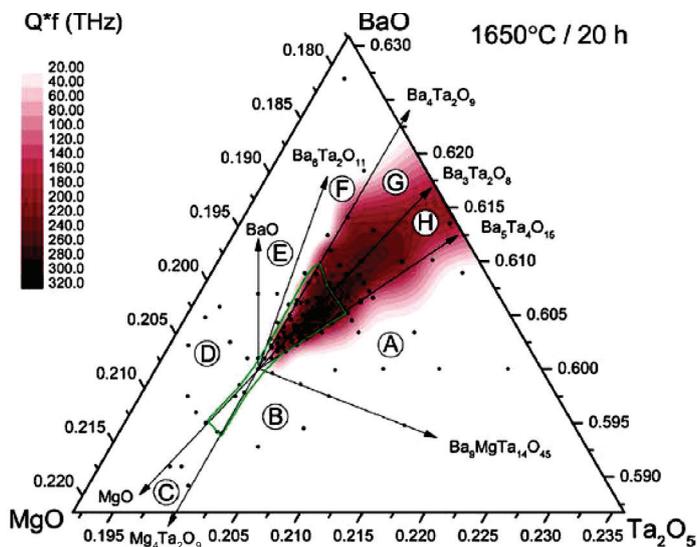


Fig. 1. Fragment of the state diagram of the BaO-MgO-Ta₂O₅ system [4]

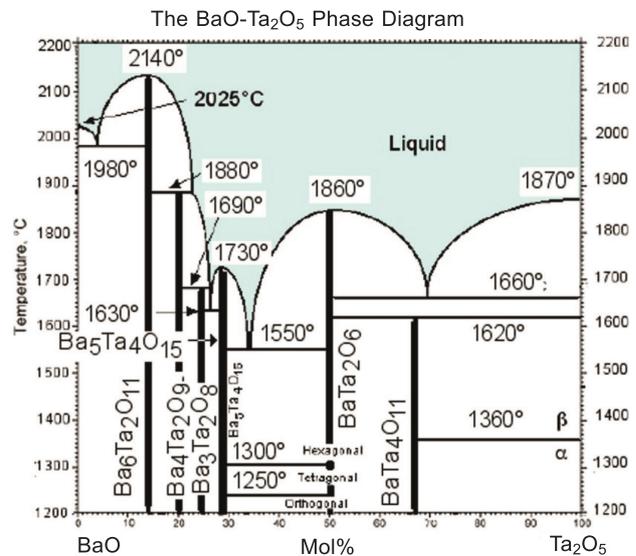


Fig. 2. State diagram of the BaO-Ta₂O₅ system

Also, the compound $Ba_3Ta_2O_{5.5}$ is noticed in [1, 7] upon the addition of B_2O_3 to $Ba(Mg_{1/3}Ta_{2/3})O_3$, when the samples are calcined at 1200 °C.

It is indicated in [8] that this compound has a cubic structure, and also disordering occurs in an orthorhombic structure with the space group Fmmm.

In [9], a related compound $Sr_6Ta_2O_{11}$ is discussed and it is noted that the transition from a cubic to an orthorhombic structure with the space symmetry group (SSG) Fmmm occurs due to hydration. Also, the conductivity of $Ba_6Ta_2O_{11}$ is measured by impedance spectroscopy [10].

The results of the analysis of the literature indicate that the compound under study has several polymorphic modifications [2], the crystal structure of which has not been sufficiently studied. By its electrical properties, it can be used as a microwave dielectric.

5. Methods of research

Diffraction spectra of compounds for the study were generated using the HighScorePlus 3.0 program and the attached PDF-2 database for 2004 in UDF format.

The analysis of the proposed structural model of this spectrum was carried out using the HighScorePlus 3.0 software by the Rietveld method.

6. Research results

Diffraction spectrum of compound $Ba_6Ta_2O_{11}$ numbered 1 in Table 1 is indexed in the orthorhombic system with lattice periods $a=6.218$ Å°; $b=8.509$ Å°; $c=6.227$ Å°. The space group of symmetry $P2_12_12_1$ is possible (19).

The correct system of points and their coordinates for a given spectrum are specified in Table 2.

The interplanar distances and integral intensities of the diffraction spectra observed and calculated for the phase are given in Table 3.

The values of interatomic distances for the compound $Ba_6Ta_2O_{11}$ numbered 1 in the Table 1 calculated by the structural model and given in Table 4.

Table 2

Microstructural parameters of Ba₆Ta₂O₁₁ for spectrum 00-049-0899 in the PDF-2 database up to 2004

| Atom | Wyck. | s.o.f. | x | y | z | U_{iso}^a |
|------|-------|----------|-----------|-----------|-----------|-------------|
| Ba1 | 4a | 1.000000 | 0.090587 | -0.007633 | 0.082019 | 4.806933 |
| Ba2 | 4a | 0.774000 | 0.274773 | -0.114156 | -0.024582 | 3.104173 |
| Ba3 | 4a | 1.000000 | -0.049231 | 0.020238 | 0.046000 | 0.620282 |
| Ba4 | 4a | 1.000000 | -0.049739 | -0.260606 | -0.046742 | 0.394535 |
| Ba5 | 4a | 0.914143 | 0.079644 | -0.231196 | -0.059195 | 1.916451 |
| Ba6 | 4a | 0.486292 | 0.246502 | -0.011633 | -0.229980 | 1.882584 |
| Ba7 | 4a | 0.825643 | -0.032989 | 0.247469 | -0.040402 | 1.284048 |
| Ta1 | 4a | 1.000000 | 0.503732 | 0.513886 | 0.542692 | 1.396722 |
| Ta2 | 4a | 0.513000 | 0.207272 | 0.322148 | 0.237971 | 4.483469 |
| Ta3 | 4a | 0.487181 | 0.300500 | 0.317155 | 0.765045 | 4.244265 |
| O1 | 4a | 1.000000 | -0.149414 | 0.780304 | 0.553505 | 0.500000 |
| O2 | 4a | 1.000000 | 0.634525 | -0.025665 | -0.112654 | 0.500000 |
| O3 | 4a | 0.500000 | 0.468125 | 0.249244 | -0.414674 | 0.500000 |
| O4 | 4a | 1.000000 | 0.838651 | -0.063485 | 0.429653 | 0.500000 |
| O5 | 4a | 1.000000 | 0.286900 | 0.326182 | 0.025695 | 0.500000 |
| O6 | 4a | 1.000000 | 0.065386 | 0.394639 | 1.200774 | 0.500000 |
| O7 | 4a | 1.000000 | 0.791818 | 0.636994 | 0.829002 | 0.500000 |
| O8 | 4a | 1.000000 | 0.688139 | 0.953246 | -0.097689 | 0.500000 |
| O9 | 4a | 1.000000 | 0.791818 | 0.636994 | 0.829002 | 0.500000 |
| O10 | 4a | 0.500000 | 0.456135 | 0.334908 | 0.264957 | 0.500000 |
| O11 | 4a | 1.000000 | 0.290817 | 0.560097 | 0.251081 | 0.500000 |
| O12 | 4a | 1.000000 | 0.149415 | 0.280313 | -0.053508 | 0.500000 |

Note: Wyck. – correct point system; s.o.f. – filling factor of positions with atoms; x, y, z – coordinates of atoms in the face of lattice periods ($x=a$; $y=b$; $z=c$); U_{iso}^a – temperature factor

Table 3

The values of interplanar distances and intensities, which are observed and calculated for the α -phase

| d_{cal} (Å°) | d_{obs} (Å°) | I_{cal} | I_{obs} | H | K | L | d_{cal} (Å°) | d_{obs} (Å°) | I_{cal} | I_{obs} | H | K | L |
|----------------|----------------|-----------|-----------|---|---|---|----------------|----------------|-----------|-----------|----|----|----|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| 5.04 | 5.04 | 35.2 | 40.7 | 0 | 1 | 1 | 1.92 | – | 0.38 | – | 3 | 1 | 1 |
| 5.03 | – | 14.7 | – | 1 | 1 | 0 | 1.92 | – | 0.21 | – | 1 | 4 | 1 |
| 4.41 | 4.41 | 10.8 | 10.3 | 1 | 0 | 1 | 1.87 | – | 0.35 | – | 0 | 2 | 3 |
| 4.26 | – | 0.79 | – | 0 | 2 | 0 | 1.86 | – | 0.30 | – | 3 | 2 | 0 |
| 3.91 | – | 1.52 | – | 1 | 1 | 1 | 1.79 | 1.79 | 82.3 | 78.4 | 1 | 2 | 3 |
| 3.52 | – | 0.41 | – | 0 | 2 | 1 | 1.79 | – | 5.02 | – | 3 | 2 | 1 |
| 3.52 | – | 0.01 | – | 1 | 2 | 0 | 1.76 | 1.76 | 26.3 | 59.1 | 0 | 4 | 2 |
| 3.12 | 3.12 | 78.5 | 80.0 | 0 | 0 | 2 | 1.76 | – | 38.5 | – | 2 | 4 | 0 |
| 3.113 | – | 18.7 | – | 2 | 0 | 0 | 1.74 | – | 0.45 | – | 2 | 3 | 2 |
| 3.06 | 3.06 | 100 | 100 | 1 | 2 | 1 | 1.73 | – | 0.88 | – | 2 | 0 | 3 |
| 2.93 | – | 0.23 | – | 0 | 1 | 2 | 1.73 | – | 0.17 | – | 3 | 0 | 2 |
| 2.92 | – | 0.15 | – | 2 | 1 | 0 | 1.69 | – | 0.57 | – | 2 | 1 | 3 |
| 2.79 | – | 0.12 | – | 1 | 0 | 2 | 1.69 | – | 0.28 | – | 3 | 1 | 2 |
| 2.78 | – | 0.06 | – | 2 | 0 | 1 | 1.69 | – | 0.14 | – | 1 | 4 | 2 |
| 2.65 | 2.65 | 10.1 | 10.1 | 1 | 1 | 2 | 1.69 | – | 0.49 | – | 2 | 4 | 1 |
| 2.65 | – | 0.31 | – | 2 | 1 | 1 | 1.68 | – | 0.40 | – | 0 | 3 | 3 |
| 2.58 | – | 2.80 | – | 0 | 3 | 1 | 1.68 | – | 1.08 | – | 3 | 3 | 0 |
| 2.58 | 2.58 | 7.57 | 10.1 | 1 | 3 | 0 | 1.64 | – | 0.18 | – | 0 | 5 | 1 |
| 2.51 | – | 0.34 | – | 0 | 2 | 2 | 1.64 | – | 0.49 | – | 1 | 5 | 0 |
| 2.51 | – | 0.56 | – | 2 | 2 | 0 | 1.62 | – | 0.16 | – | 1 | 3 | 3 |
| 2.39 | – | 0.92 | – | 1 | 3 | 1 | 1.62 | – | 1.40 | – | 3 | 3 | 1 |
| 2.33 | – | 0.52 | – | 1 | 2 | 2 | 1.60 | – | 0.13 | – | 2 | 2 | 3 |
| 2.33 | – | 0.33 | – | 2 | 2 | 1 | 1.60 | – | 0.09 | – | 3 | 2 | 2 |
| 2.20 | 2.21 | 70.8 | 59.2 | 2 | 0 | 2 | 1.59 | – | 0.35 | – | 1 | 5 | 1 |
| 2.13 | – | 1.19 | – | 2 | 1 | 2 | 1.56 | 1.56 | 6.62 | 9.95 | 0 | 0 | 4 |

Continuation of Table 3

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|-------|------|------|------|---|---|---|------|------|------|------|----|----|----|
| 2.13 | 2.13 | 44.5 | 39.5 | 0 | 4 | 0 | 1.56 | – | 4.92 | – | 4 | 0 | 0 |
| 2.09 | – | 0.32 | – | 0 | 3 | 2 | 1.53 | – | 0.05 | – | 0 | 1 | 4 |
| 2.10 | – | 0.59 | – | 2 | 3 | 0 | 1.53 | 1.53 | 40.2 | 39.6 | 2 | 4 | 2 |
| 2.02 | – | 0.80 | – | 0 | 1 | 3 | 1.53 | – | 1.00 | – | 4 | 1 | 0 |
| 2.01 | – | 0.59 | – | 3 | 3 | 0 | 1.51 | – | 0.08 | – | 1 | 0 | 4 |
| 2.01 | – | 0.24 | – | 0 | 4 | 1 | 1.51 | – | 1.33 | – | 4 | 0 | 1 |
| 2.01 | – | 0.03 | – | 1 | 4 | 0 | 1.49 | – | 0.17 | – | 0 | 5 | 2 |
| 1.99 | – | 14.5 | – | 1 | 3 | 2 | 1.49 | – | 0.56 | – | 2 | 5 | 0 |
| 1.99 | 1.99 | 23.8 | 39.6 | 2 | 2 | 1 | 1.49 | – | 1.23 | – | 1 | 1 | 4 |
| 1.97 | – | 0.13 | – | 1 | 0 | 3 | 1.49 | – | 0.11 | – | 0 | 4 | 3 |
| 1.97 | – | 0.81 | – | 3 | 0 | 1 | 1.49 | – | 0.26 | – | 4 | 1 | 1 |
| 1.96 | – | 0.14 | – | 2 | 2 | 2 | 1.48 | – | 0.12 | – | 3 | 4 | 0 |
| 1.927 | – | 0.41 | – | 1 | 1 | 3 | | | | | | | |

Note: odds factor $R=8.54707\%$; d_{cal} – interplanar distance calculated according to the structural model; d_{obs} – interplanar distance, observed (found by the Wolfe-Bragg formula); I_{cal} – integrated intensity of the diffraction maximum, calculated for the structural model; I_{obs} – integrated intensity of the diffraction maximum of the studied spectrum of the compound; H, K, L – Miller indices

Table 4

 $Ba_6Ta_2O_{11}$ interatomic distances according to the proposed structural model of the α -phase

| Atom1 | Atom2 | Distance, A° | Atom1 | Atom2 | Distance, A° | Atom1 | Atom2 | Distance, A° |
|-------|-------|--------------|-------|-------|--------------|-------|-------|--------------|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| Ba1 | –Ba3 | 0.929 | Ta1 | –O2 | 0.982 | O7 | –Ba2 | 1.238 |
| – | –Ba6 | 1.557 | – | –O4 | 1.123 | – | –Ba6 | 1.427 |
| – | –Ba2 | 1.604 | – | –O6 | 1.327 | – | –Ba4 | 1.526 |
| – | –O7 | 1.756 | – | –O8 | 1.344 | – | –O10 | 1.635 |
| – | –O6 | 1.861 | – | –Ba2 | 1.760 | – | –Ta3 | 1.739 |
| – | –O2 | 1.942 | – | –Ba6 | 1.954 | – | –Ba1 | 1.756 |
| – | –O3 | 2.088 | – | –O12 | 2.081 | – | –O3 | 1.952 |
| – | –Ba5 | 2.097 | – | –Ba7 | 2.103 | – | –O2 | 2.056 |
| – | –O2 | 2.101 | – | –O7 | 2.244 | – | –Ba5 | 2.107 |
| – | –Ba6 | 2.172 | – | –Ba4 | 2.244 | – | –O1 | 2.136 |
| – | –Ta1 | 2.399 | – | –O5 | 2.265 | – | –O2 | 2.165 |
| – | –Ba7 | 2.426 | – | –Ta2 | 2.269 | – | –Ba5 | 2.224 |
| – | –Ba4 | 2.457 | – | –O3 | 2.278 | – | –Ta1 | 2.244 |
| – | –O5 | 2.530 | – | –O11 | 2.281 | – | –O6 | 2.375 |
| – | –Ta2 | 2.605 | – | –O10 | 2.323 | – | –O3 | 2.390 |
| – | –Ta3 | 2.605 | – | –O11 | 2.331 | – | –Ba7 | 2.522 |
| – | –O12 | 2.617 | – | –Ba1 | 2.399 | – | –O11 | 2.625 |
| – | –Ta1 | 2.646 | – | –Ba6 | 2.464 | – | –Ba3 | 2.625 |
| – | –O11 | 2.653 | – | –Ta3 | 2.513 | – | –Ba6 | 2.730 |
| – | –O8 | 2.683 | – | –Ba5 | 2.541 | – | –Ba4 | 2.730 |
| – | –O4 | 2.714 | – | –Ba3 | 2.599 | – | –Ta1 | 2.736 |
| – | –O10 | 2.744 | – | –Ba1 | 2.646 | – | –O5 | 2.777 |
| – | –O8 | 2.761 | – | –O7 | 2.736 | – | –O8 | 2.805 |
| – | –Ba2 | 2.789 | – | –Ba3 | 2.880 | – | –Ba2 | 2.887 |
| – | –O11 | 2.824 | – | –Ta3 | 2.938 | – | –Ba1 | 2.933 |
| – | –O4 | 2.896 | – | –O5 | 2.990 | – | –Ba7 | 2.975 |
| – | –O7 | 2.933 | – | –O3 | 3.067 | – | –Ba3 | 3.000 |
| – | –Ta3 | 2.949 | – | –Ta3 | 3.094 | – | –O8 | 3.076 |
| – | –Ba4 | 2.990 | – | –Ta2 | 3.108 | – | –Ta2 | 3.123 |
| – | –Ta2 | 3.057 | – | –O1 | 3.130 | – | –O11 | 3.128 |

Continuation of Table 4

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|----|------|-------|-----|------|-------|-----|------|-------|
| - | -05 | 3.111 | - | -Ba5 | 3.327 | - | -Ta2 | 3.174 |
| - | -03 | 3.115 | - | -010 | 3.346 | - | -Ta2 | 3.185 |
| - | -Ba7 | 3.232 | - | -08 | 3.407 | - | -04 | 3.232 |
| - | -010 | 3.263 | - | -Ta2 | 3.412 | - | -05 | 3.245 |
| - | -01 | 3.361 | - | -010 | 3.427 | - | -04 | 3.305 |
| - | -010 | 3.426 | - | -Ba3 | 3.437 | - | -010 | 3.407 |
| - | -Ta2 | 3.485 | - | -02 | 3.499 | 011 | -08 | 1.155 |
| 04 | -08 | 0.968 | Ta2 | -06 | 1.101 | - | -Ta3 | 1.192 |
| - | -06 | 1.069 | - | -05 | 1.412 | - | -04 | 1.738 |
| - | -Ta1 | 1.123 | - | -04 | 1.455 | - | -012 | 1.861 |
| - | -05 | 1.252 | - | -010 | 1.560 | - | -Ba3 | 1.992 |
| - | -Ta2 | 1.455 | - | -012 | 1.884 | - | -04 | 2.011 |
| - | -01 | 1.539 | - | -03 | 1.947 | - | -06 | 2.011 |
| - | -011 | 1.738 | - | -Ba5 | 1.987 | - | -05 | 2.023 |
| - | -Ba3 | 1.974 | - | -011 | 2.092 | - | -Ta2 | 2.092 |
| - | -02 | 2.007 | - | -08 | 2.105 | - | -010 | 2.176 |
| - | -011 | 2.011 | - | -Ba7 | 2.115 | - | -Ba3 | 2.209 |
| - | -Ba7 | 2.127 | - | -Ta1 | 2.269 | - | -Ta1 | 2.281 |
| - | -Ta3 | 2.323 | - | -Ba4 | 2.270 | - | -Ta1 | 2.331 |
| - | -010 | 2.361 | - | -Ba2 | 2.309 | - | -08 | 2.346 |
| - | -Ta3 | 2.433 | - | -Ba3 | 2.370 | - | -Ba4 | 2.348 |
| - | -Ba7 | 2.469 | - | -Ba7 | 2.375 | - | -05 | 2.436 |
| - | -Ba3 | 2.589 | - | -Ba4 | 2.570 | - | -03 | 2.508 |
| - | -Ba1 | 2.714 | - | -08 | 2.586 | - | -Ba7 | 2.576 |
| - | -03 | 2.794 | - | -Ba1 | 2.605 | - | -Ba5 | 2.602 |
| - | -010 | 2.817 | - | -Ba6 | 2.665 | - | -07 | 2.625 |
| - | -Ba6 | 2.837 | - | -Ba3 | 2.687 | - | -Ba1 | 2.653 |
| - | -Ba2 | 2.854 | - | -Ba5 | 2.718 | - | -02 | 2.797 |
| - | -Ba1 | 2.896 | - | -02 | 2.727 | - | -010 | 2.820 |
| - | -03 | 2.922 | - | -03 | 2.774 | - | -Ba1 | 2.824 |
| - | -Ba4 | 2.983 | - | -Ta3 | 2.793 | - | -Ba6 | 2.866 |
| - | -Ba5 | 2.984 | - | -02 | 2.850 | - | -Ba7 | 2.891 |
| - | -Ta2 | 3.028 | - | -Ta3 | 3.002 | - | -Ba5 | 2.934 |
| - | -08 | 3.092 | - | -04 | 3.028 | - | -06 | 2.965 |
| - | -06 | 3.201 | - | -Ba1 | 3.057 | - | -03 | 3.037 |
| - | -07 | 3.232 | - | -Ta3 | 3.074 | - | -Ba7 | 3.126 |
| - | -Ta3 | 3.278 | - | -01 | 3.104 | - | -07 | 3.128 |
| - | -05 | 3.283 | - | -Ta1 | 3.108 | - | -Ba5 | 3.143 |
| - | -Ba4 | 3.294 | - | -07 | 3.123 | - | -012 | 3.168 |
| - | -07 | 3.305 | - | -07 | 3.174 | - | -02 | 3.189 |
| - | -Ba5 | 3.312 | - | -07 | 3.185 | - | -Ta2 | 3.193 |
| - | -Ba6 | 3.334 | - | -011 | 3.193 | - | -Ba4 | 3.202 |
| - | -010 | 3.365 | - | -Ba3 | 3.252 | - | -Ba2 | 3.262 |
| - | -08 | 3.418 | - | -Ba6 | 3.288 | - | -Ba4 | 3.267 |
| - | -04 | 3.475 | - | -Ta3 | 3.333 | - | -011 | 3.316 |
| - | -Ba4 | 3.478 | - | -05 | 3.335 | - | -Ta2 | 3.349 |
| - | -05 | 3.496 | - | -011 | 3.349 | - | -Ba2 | 3.355 |
| - | - | - | - | -Ta1 | 3.412 | - | -Ba6 | 3.412 |
| - | - | - | - | -Ba1 | 3.485 | - | -03 | 3.463 |
| - | - | - | - | -05 | 3.488 | - | - | - |

Diffraction spectrum of compound number 3 in the Table 1 is indexed in the orthorhombic system. The space group of symmetry Fmmm (69) with lattice periods $a=8.668(7)$ Å° is possible; $b=8.677(8)$ Å°; $c=8.685(7)$ Å°. The values of the microstructural parameters are given in Table 5.

The values of interplanar distances and integrated intensities of the diffraction spectra observed and calculated are given in Table 6.

The values of the interatomic distances for this structural model are given in Table 7.

Table 5

Microstructural parameters of Ba₆Ta₂O₁₁ for spectrum 00-049-0903 in the PDF-2 database up to 2004

| Atom | Wyck. | s.o.f. | x | y | z | U_{iso}^a |
|------|-------|----------|----------|----------|----------|-------------|
| Ba1 | 4a | 0.500000 | 0.000000 | 0.000000 | 0.000000 | 0.500000 |
| Ba2 | 8f | 0.500000 | 0.250000 | 0.250000 | 0.750000 | 0.500000 |
| Ta1 | 4b | 0.500000 | 0.500000 | 0.500000 | 0.500000 | 0.500000 |
| O1 | 32p | 0.687500 | -0.38(3) | 0.11(4) | 0.900000 | 0.500000 |

Note: Wyck. – correct point system; s.o.f. – filling factor of positions with atoms; x, y, z – coordinates of atoms in the face of lattice periods ($x=a$; $y=b$; $z=c$); U_{iso}^a – temperature factor

Table 6

The values of the interplanar distances and intensities are observed and calculated for the β -phase of the compound

| d_{cal} (Å°) | d_{obs} (Å°) | I_{cal} | I_{obs} | H | K | L | d_{cal} (Å°) | d_{obs} (Å°) | I_{cal} | I_{obs} | H | K | L |
|----------------|----------------|-----------|-----------|---|---|---|----------------|----------------|-----------|-----------|---|---|---|
| 5.00 | 5.01 | 8.76 | 20.3 | 1 | 1 | 1 | 1.53 | 1.53 | 12.6 | 30.3 | 0 | 4 | 4 |
| 4.34 | 4.34 | 11.9 | 20.3 | 0 | 0 | 2 | 1.53 | – | 12.7 | – | 4 | 0 | 4 |
| 4.33 | – | 0.35 | – | 0 | 2 | 0 | 1.53 | – | 5.68 | – | 4 | 4 | 0 |
| 4.33 | – | 0.22 | – | 2 | 0 | 0 | 1.47 | – | 0.47 | – | 1 | 3 | 5 |
| 3.07 | 3.07 | 90.6 | 100.0 | 0 | 2 | 2 | 1.47 | – | 0.58 | – | 3 | 1 | 5 |
| 3.07 | – | 89.5 | – | 2 | 0 | 2 | 1.47 | – | 0.11 | – | 1 | 5 | 3 |
| 3.06 | – | 38.7 | – | 2 | 2 | 0 | 1.47 | – | 0.14 | – | 3 | 5 | 1 |
| 2.62 | – | 0.46 | – | 1 | 1 | 3 | 1.46 | – | 0.10 | – | 5 | 1 | 3 |
| 2.61 | – | 0.02 | – | 1 | 3 | 1 | 1.46 | – | 0.11 | – | 5 | 3 | 1 |
| 2.61 | – | 0.02 | – | 3 | 1 | 1 | 1.44 | – | 0.06 | – | 0 | 0 | 6 |
| 2.50 | – | 0.32 | – | 2 | 2 | 2 | 1.44 | – | 0.09 | – | 2 | 4 | 4 |
| 2.17 | 2.17 | 100.0 | 60.1 | 0 | 0 | 4 | 1.44 | – | 0.00 | – | 0 | 6 | 0 |
| 2.17 | – | 5.17 | – | 0 | 4 | 0 | 1.44 | – | 0.11 | – | 4 | 2 | 4 |
| 2.17 | – | 5.09 | – | 4 | 0 | 0 | 1.44 | – | 0.10 | – | 4 | 4 | 2 |
| 1.99 | – | 0.36 | – | 1 | 3 | 3 | 1.44 | – | 0.01 | – | 6 | 0 | 0 |
| 1.99 | – | 0.41 | – | 3 | 1 | 3 | 1.37 | 1.37 | 26.7 | 30.4 | 0 | 2 | 6 |
| 1.99 | – | 0.30 | – | 3 | 3 | 1 | 1.37 | – | 27.1 | – | 2 | 0 | 6 |
| 1.94 | – | 0.16 | – | 0 | 2 | 4 | 1.37 | – | 3.34 | – | 0 | 6 | 2 |
| 1.94 | – | 0.24 | – | 2 | 0 | 4 | 1.37 | – | 3.00 | – | 2 | 6 | 0 |
| 1.94 | – | 0.01 | – | 0 | 4 | 2 | 1.37 | – | 3.40 | – | 6 | 0 | 2 |
| 1.94 | – | 0.04 | – | 2 | 4 | 0 | 1.37 | – | 2.99 | – | 6 | 2 | 0 |
| 1.94 | – | 0.01 | – | 4 | 0 | 2 | 1.32 | – | 0.02 | – | 3 | 3 | 5 |
| 1.94 | – | 0.03 | – | 4 | 2 | 0 | 1.32 | – | 0.02 | – | 3 | 5 | 3 |
| 1.77 | 1.77 | 52.57 | 80.3 | 2 | 2 | 4 | 1.32 | – | 0.02 | – | 5 | 3 | 3 |
| 1.77 | – | 18.60 | – | 2 | 4 | 2 | 1.31 | – | 0.16 | – | 2 | 2 | 6 |
| 1.77 | – | 18.40 | – | 4 | 2 | 2 | 1.31 | – | 0.03 | – | 2 | 6 | 2 |
| 1.67 | – | 0.06 | – | 1 | 1 | 5 | 1.31 | – | 0.03 | – | 6 | 2 | 2 |
| 1.67 | – | 0.01 | – | 1 | 5 | 1 | | | | | | | |
| 1.67 | – | 0.06 | – | 3 | 3 | 3 | | | | | | | |
| 1.67 | – | 0.01 | – | 5 | 1 | 1 | | | | | | | |

Note: odds factor $H=7.03646$ %. d_{cal} – interplanar distance calculated according to the structural model; d_{obs} – interplanar distance, observed (found by the Wolfe-Bragg formula); I_{cal} – integrated intensity of the diffraction maximum, calculated for the structural model; I_{obs} – integrated intensity of the diffraction maximum of the studied spectrum of the compound; H, K, L – Miller indices

Table 7

The values of the interatomic distances of the crystal structure of the β -phase of the compound

| Atom1 | Atom2 | Distance, A° | Atom1 | Atom2 | Distance, A° | Atom1 | Atom2 | Distance, A° |
|-------|-------|--------------|-------|-------|--------------|-------|-------|--------------|
| Ta1 | -01 | 1.671 | 01 | -Ta1 | 1.671 | Ba2 | -01 | 2.093 |
| - | - | - | - | -01 | 1.737 | - | -01 | 3.453 |
| - | - | - | - | -01 | 2.006 | - | - | - |
| - | - | - | - | -01 | 2.033 | - | - | - |
| - | - | - | - | -Ba2 | 2.093 | - | - | - |
| - | - | - | - | -01 | 2.653 | - | - | - |
| - | - | - | - | -01 | 2.674 | - | - | - |
| - | - | - | - | -01 | 2.856 | - | - | - |
| - | - | - | - | -01 | 3.276 | - | - | - |
| - | - | - | - | -01 | 3.343 | - | - | - |
| - | - | - | - | -Ba2 | 3.453 | - | - | - |
| - | - | - | - | -01 | 3.476 | - | - | - |
| - | - | - | - | -01 | 3.497 | - | - | - |

Thus, the structure of the α -phase of the compound is completely occupied by the positions of the B2, B5, B6, B7, Ta2, Ta3, O3, O10 atoms (Table 2). The structure of the β -phase has regular systems of points 4a, 4b, 8f, 32p, completely filled with atoms (Table 5). This leads to distortion of the structure.

7. SWOT analysis of research results

Strengths. As a result of research, structural models are proposed for the diffraction spectra of the $\text{Ba}_6\text{Ta}_2\text{O}_{11}$ compound numbered 1 and 3 in Table 1. Both structural models belong to the orthorhombic system. Space group $P2_12_12_1$ (19) has 2-order helical symmetry axes parallel to the x, y, z axes. The space group of symmetry Fmmm (69) has specular reflection planes perpendicular to the x, y, z axes. They have elements of symmetry: the axis of symmetry is 2 orders parallel to the axes x, y, z . Also, the helical symmetry axes of the 2nd order are parallel to the x, y, z axes and the grazing reflection plane are perpendicular to the x, y, z axes with n -slip along the diagonal. The conducted studies of the crystal structure of the compound allow to study in more detail its physical properties, in particular, as a microwave dielectric.

Weaknesses. Polymorphic modifications of this compound belong to the orthorhombic system, and not to the tetragonal one, as it was indicated and indexed in [2]. Disordering also occurs from cubic to rhombic systems.

Opportunities. A polymorphic transformation from the α -phase to the β -phase at a temperature of 540 °C is recorded. The formation of one or another polymorphic modification of a compound is influenced by the method of its preparation. It is also indicated that this compound may have metastable phases.

Threats. Phase transitions of this compound are associated not only with a change in the crystal symmetry (the appearance of new symmetry elements), but also with the existence of new defects in the filling of the positions of atoms in the structure. Thus, the β -phase of the compound has completely filled correct systems of points for all atoms of the structure (Table 5).

8. Conclusions

1. Using the TREOR program, indexing the X-ray diffraction patterns of the $\text{Ba}_6\text{Ta}_2\text{O}_{11}$ compound under the numbers 00-049-0899 and in the PDF-2 database for 2004 (Table 1). The diffraction spectrum of the α -phase of compound 00-049-0899 is indexed in the orthorhombic system with lattice periods $a=6.218$ A°; $b=8.509$ A; $c=6.227$ A°. The diffraction spectrum of the β -phase (compound 00-049-0903) is indexed in the orthorhombic system with lattice periods $a=8.668(7)$ A°; $b=8.677(8)$ A°; $c=8.685(7)$ A°.

2. It is shown that the space group of symmetry of the α -phase of the compound 00-049-0899: $P2_12_12_1$ (19) is possible and a structural model is proposed for calculation.

It is also shown that the space group of symmetry of the β -phase of the compound 00-049-0903: Fmmm is possible (69), and a structural model is proposed for calculation.

3. Using the HighScorePlus 3.0 software, the parameters of the structural models of the α - and β -phases of the compound under study are refined by the Rietveld method. Microstructural parameters are given in Table 2 and Table 5, respectively.

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