

# Investigation of multiphase equilibria in the subsolidus of BaO–CoO–Fe<sub>2</sub>O<sub>3</sub>–Al<sub>2</sub>O<sub>3</sub> system

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**Abstract.** One of the most important problems related to the development of new nonmetal materials and their performance characteristics is to predict the phase composition. The most comprehensive information on phase interactions and the thermodynamic stability of phase combinations is given by the state diagrams. The materials synthesized in the system subsolidus domain can be predicted the most accurately, because their sintering occurs without participation of the melt. Due to the above fact, the studies of the subsolidus structure of BaO – CoO – Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system are of great interest, because on the basis of this system we can obtain a huge amount of nonmetal materials with prescribed properties, for example ferrimagnetic materials to protect from electromagnetic radiation, because the system compounds have cementing, refractory and ferrimagnetic properties. To study the structure of BaO – CoO – Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system in detail the authors summed up already known data on the thermodynamic constants of system compounds. This allowed us to do the thermodynamic analysis of multiphase equilibrium processes that occur in the subsolidus of BaO – CoO – Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system that was used as a basis for the plotting of the state diagram for the subsolidus domain of the system. A promising field for the application of obtained data is the cement production technology. The produced cement can be used independently and as a binding material to produce special cements and materials that retain their properties when exposed to the action of high-frequency electromagnetic radiation.

## 1 Introduction

Prediction of the phase composition is one of the most important problems for the development of new types of refractory nonmetal materials and the conditions for their use. The most complete information about phase interrelations and the thermodynamic stability of phase combinations is given in the state diagrams in which thermodynamically

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equilibrium compositions are interconnected with the temperature. The BaO – CoO – Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system is of great interest for the researchers from the standpoint of the possibility of the production of ferrimagnetic materials that protect from the electromagnetic radiation, because its compounds have cementing, refractory and ferromagnetic properties.

However, the unavailability of data on the structure of the above system hampers the development of special materials with prescribed properties. Therefore, this scientific paper delves into the studies of the structure of BaO – CoO – Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system and solid-phase reactions in it.

## 2 Analysis of the latest research papers and publications

The BaO – CoO – Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system has a rather complicated structure that has not been properly studied yet.

The data on the structure of binary systems of refractory oxides were summarized in the references based on computational and experimental data [1].

The Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system was studied by the authors [2]. The obtained data show that it contains the hematite Fe<sub>2</sub>O<sub>3</sub>, corundum Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>Al<sub>2</sub>O<sub>6</sub>. The last compound is stable in the air right up to the temperature of 1410°C. At a higher temperature it is decomposed into corundum and the spinel-like solid solution.

The CoO – Al<sub>2</sub>O<sub>3</sub> system was studied in the subsolidus domain [3] at a temperature of 1400°C. CoAl<sub>2</sub>O<sub>4</sub> is the only stable compound in it.

According to the data obtained by the author [4] the CoO – Fe<sub>2</sub>O<sub>3</sub> is thermally stable right up to the temperature of 1200°C and they established the existence of one compound in it, in particular cobalt ferrite CoFe<sub>2</sub>O<sub>4</sub>.

The authors of scientific papers [5-8] gave consideration to CoO – BaO – Al<sub>2</sub>O<sub>3</sub>, CoO – BaO – Fe<sub>2</sub>O<sub>3</sub>, CoO – Al<sub>2</sub>O<sub>3</sub> – Fe<sub>2</sub>O<sub>3</sub>, and BaO – Al<sub>2</sub>O<sub>3</sub> – Fe<sub>2</sub>O<sub>3</sub> systems. Their thermodynamic and geometro-topological characteristics were given and that allows us to proceed to more detailed studies of CoO – BaO – Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system.

## 3 Defining research objectives and problems

The data of tetrahedration of these systems give important information on the prediction of the direction (vector) of chemical interactions and phase combinations for four-component physical and chemical systems synthesized in the products of solid phase reaction. In the tetrahedrated system connodas combine coexisting equilibrium phases by pairs; compound composition points located at the apexes of elementary triangles consolidate triple equilibrium combinations of the phases; the apexes of elementary tetrahedrons correspond to the points of compound compositions that can coexist in the four-phase equilibrium combinations. Therefore, the main problem on the determination of the subsolidus structure for four-component systems is related to the fragmentation of the concentrated tetrahedron of the system into elementary triangles, i.e. tetrahedration.

Based on the above, we arrived at a conclusion that it is topical to study the problem on multiphase equilibrium processes that occur in the subsolidus of BaO – CoO – Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system. The set goal can be achieved based on the thermodynamic analysis done for all possible combinations of the phases, peculiar for the above system.

## 4 Core of the Research

To do a theoretical research for the BaO – CoO – Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system it is reasonable to carry out the thermodynamic analysis of the processes that occur in it. This can be done, if initial thermodynamic constants are available. Table 1 gives the initial data for the thermodynamic analysis.

**Table 1.** Initial thermodynamic data

Compound formula	-ΔH <sup>0</sup> <sub>298</sub> , kJ/mole	S <sup>0</sup> <sub>298</sub> , J/mole·K	C <sub>p</sub> =a+b·T+c·T <sup>-2</sup> , J/mole·K		
			a	b·10 <sup>3</sup>	-c·10 <sup>-5</sup>
CoO	238,9	52,97	48,28	8,535	1,67
BaO	558,15	70,29	53,30	4,35	8,3
Fe <sub>2</sub> O <sub>3</sub>	821,36	89,96	98,28	9,04	14,85
α-Al <sub>2</sub> O <sub>3</sub>	1675,61	50,92	114,77	12,08	35,44
CoAl <sub>2</sub> O <sub>4</sub>	1948,9	101,7	161,17	32,97	30,32
CoFe <sub>2</sub> O <sub>4</sub>	1087,4	134,7	131,8	141,42	–
Fe <sub>2</sub> Al <sub>2</sub> O <sub>6</sub>	1262,73	71,55	50,84	6,54	21,93
BaFe <sub>12</sub> O <sub>19</sub>	5886,09	609,19	348,60	1168,82	–
BaFe <sub>2</sub> O <sub>4</sub>	1282,81	158,99	172,38	20,88	5,94
Ba <sub>2</sub> Fe <sub>2</sub> O <sub>5</sub>	1763,97	229,07	295,03	–	51,09
Ba <sub>3</sub> Fe <sub>2</sub> O <sub>6</sub>	2620,61	312,63	219,03	90,80	14,48
Ba <sub>5</sub> Fe <sub>2</sub> O <sub>8</sub>	3635,56	809,76	288,11	150,90	6,05
Ba <sub>7</sub> Fe <sub>4</sub> O <sub>13</sub>	5541,71	687,26	506,88	112,10	37,99
Ba <sub>2</sub> Fe <sub>6</sub> O <sub>11</sub>	3752,80	437,4	398,94	119,50	59,91
BaCoO <sub>2</sub>	837,13	123,08	78,99	38,34	4,49
BaAl <sub>2</sub> O <sub>4</sub>	2334,17	123,43	148,32	35,44	29,25
Ba <sub>3</sub> Al <sub>2</sub> O <sub>6</sub>	3537,91	267,78	247,86	48,53	17,41
BaAl <sub>12</sub> O <sub>19</sub>	10740,33	376,56	738,22	70,50	221,75
Ba <sub>4</sub> Al <sub>2</sub> O <sub>7</sub>	4014,49	329,99	275,85	56,89	23,33
Ba <sub>5</sub> Al <sub>2</sub> O <sub>8</sub>	4569,03	427,82	298,73	123,87	15,51
Ba <sub>7</sub> Al <sub>2</sub> O <sub>10</sub>	5689,5	567,66	374,18	164,90	14,03
Ba <sub>8</sub> Al <sub>2</sub> O <sub>11</sub>	6238,10	611,68	441,99	96,23	25,31
Ba <sub>10</sub> Al <sub>2</sub> O <sub>13</sub>	7350,52	778,85	478,25	226,40	12,24
Ba <sub>2</sub> Co <sub>2</sub> Fe <sub>12</sub> O <sub>22</sub>	6848,23	785,98	812,71	203,71	140,65
BaCo <sub>2</sub> Fe <sub>16</sub> O <sub>27</sub>	7987,63	895,36	1029,37	151,06	214,45
Ba <sub>3</sub> Co <sub>2</sub> Fe <sub>24</sub> O <sub>41</sub>	12609,45	1396,22	1566,51	255,75	314,43

The combination of triple systems triangulated earlier [5-8] into the concentrated tetrahedron of BaO – CoO – Fe<sub>2</sub>O<sub>3</sub> – Al<sub>2</sub>O<sub>3</sub> system closes automatically elementary tetrahedrons:

- 1) BaO – Ba<sub>5</sub>Fe<sub>2</sub>O<sub>8</sub> – BaCoO<sub>2</sub> – Ba<sub>10</sub>Al<sub>2</sub>O<sub>13</sub>;
- 2) Ba<sub>5</sub>Fe<sub>2</sub>O<sub>8</sub> – BaCoO<sub>2</sub> – Ba<sub>10</sub>Al<sub>2</sub>O<sub>13</sub> – Ba<sub>7</sub>Fe<sub>4</sub>O<sub>13</sub>;
- 3) BaCoO<sub>2</sub> – Ba<sub>10</sub>Al<sub>2</sub>O<sub>13</sub> – Ba<sub>7</sub>Fe<sub>4</sub>O<sub>13</sub> – Ba<sub>3</sub>Fe<sub>2</sub>O<sub>6</sub>;
- 4) BaCoO<sub>2</sub> – Ba<sub>10</sub>Al<sub>2</sub>O<sub>13</sub> – Ba<sub>3</sub>Fe<sub>2</sub>O<sub>6</sub> – Ba<sub>8</sub>Al<sub>2</sub>O<sub>11</sub>;
- 5) BaCoO<sub>2</sub> – Ba<sub>8</sub>Al<sub>2</sub>O<sub>11</sub> – Ba<sub>3</sub>Fe<sub>2</sub>O<sub>6</sub> – Ba<sub>7</sub>Al<sub>2</sub>O<sub>10</sub>;
- 6) BaCoO<sub>2</sub> – Ba<sub>7</sub>Al<sub>2</sub>O<sub>10</sub> – Ba<sub>3</sub>Fe<sub>2</sub>O<sub>6</sub> – Ba<sub>5</sub>Al<sub>2</sub>O<sub>8</sub>;
- 7) BaCoO<sub>2</sub> – Ba<sub>5</sub>Al<sub>2</sub>O<sub>8</sub> – Ba<sub>3</sub>Fe<sub>2</sub>O<sub>6</sub> – Ba<sub>4</sub>Al<sub>2</sub>O<sub>7</sub>;
- 8) BaCoO<sub>2</sub> – Ba<sub>4</sub>Al<sub>2</sub>O<sub>7</sub> – Ba<sub>3</sub>Fe<sub>2</sub>O<sub>6</sub> – Ba<sub>3</sub>Al<sub>2</sub>O<sub>6</sub>;
- 9) BaCoO<sub>2</sub> – Ba<sub>3</sub>Al<sub>2</sub>O<sub>6</sub> – Ba<sub>3</sub>Fe<sub>2</sub>O<sub>6</sub> – BaAl<sub>2</sub>O<sub>4</sub>;
- 10) BaCoO<sub>2</sub> – BaAl<sub>2</sub>O<sub>4</sub> – Ba<sub>3</sub>Fe<sub>2</sub>O<sub>6</sub> – Ba<sub>2</sub>Fe<sub>6</sub>O<sub>11</sub>;
- 11) BaCoO<sub>2</sub> – BaAl<sub>2</sub>O<sub>4</sub> – Ba<sub>2</sub>Fe<sub>6</sub>O<sub>11</sub> – BaFe<sub>2</sub>O<sub>4</sub>;
- 12) BaCoO<sub>2</sub> – BaAl<sub>2</sub>O<sub>4</sub> – BaFe<sub>2</sub>O<sub>4</sub> – Ba<sub>2</sub>Fe<sub>6</sub>O<sub>11</sub>;
- 13) BaCoO<sub>2</sub> – BaAl<sub>2</sub>O<sub>4</sub> – Ba<sub>2</sub>Fe<sub>6</sub>O<sub>11</sub> – BaFe<sub>12</sub>O<sub>19</sub>.

The  $\text{BaCoO}_2 - \text{BaAl}_2\text{O}_4 - \text{BaFe}_{12}\text{O}_{19}$  face (the elementary tetrahedron No13) is simultaneously coincidental with the faces of the closed tetrahedron domain with the apexes of  $\text{BaCoO}_2$ ,  $\text{BaBaFe}_{12}\text{O}_{19}$  and  $\text{CoO}$ . In addition, the points of the compositions of triple compounds  $\text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$  and  $\text{Ba}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22}$  belong to one of the faces of this domain and the points of the compound compositions that allow for the partition of the domain into elementary domains are unavailable on other faces and ribs. This circumstance is indicative of the fact that the tetrahedration of specified domain is only possible due to the stability of “internal” connodas that pass through the concentration space of studied system:  $\text{BaAl}_2\text{O}_4 - \text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$ , and  $\text{BaAl}_2\text{O}_4 - \text{Ba}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22}$ . These connodas define the existence of elementary tetrahedrons:

- 14)  $\text{BaCoO}_2 - \text{BaAl}_2\text{O}_4 - \text{Ba}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22} - \text{CoO}$ ;
- 15)  $\text{BaCoO}_2 - \text{BaAl}_2\text{O}_4 - \text{Ba}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22} - \text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41}$ ;
- 16)  $\text{BaCoO}_2 - \text{BaAl}_2\text{O}_4 - \text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41} - \text{BaFe}_{12}\text{O}_{19}$ ;
- 17)  $\text{BaAl}_2\text{O}_4 - \text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41} - \text{BaFe}_{12}\text{O}_{19} - \text{CoO}$ ;
- 18)  $\text{BaAl}_2\text{O}_4 - \text{Ba}_3\text{Co}_2\text{Fe}_{24}\text{O}_{41} - \text{Ba}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22} - \text{CoO}$ .

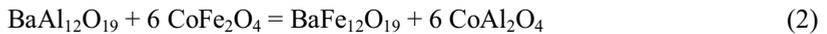
The binary compositions  $\text{BaFe}_{12}\text{O}_{19}$ ,  $\text{Fe}_2\text{Al}_2\text{O}_6$ , and  $\text{CoFe}_2\text{O}_4$  proximal to the apex of  $\text{Fe}_2\text{O}_3$  that are located on the sides of concentrated tetrahedron of the system specify the tetrahedral domain circumscribed by appropriate points of the compositions of these compounds. In addition, the connoda  $\text{Ba}_2\text{Fe}_{12}\text{O}_{19} - \text{CoFe}_2\text{O}_4$  also has a point of the composition of triple compound  $\text{Ba}_2\text{Co}_2\text{Fe}_{16}\text{O}_{27}$  that coexists with  $\text{Fe}_2\text{O}_3$  due to the availability of  $\text{Fe}_2\text{O}_3 - \text{Ba}_2\text{Co}_2\text{Fe}_{16}\text{O}_{27}$ . The only option for the partition of tetrahedral domain (circumscribed by the points of  $\text{Fe}_2\text{O}_3$ ,  $\text{BaBaFe}_{12}\text{O}_{19}$ ,  $\text{Fe}_2\text{Al}_2\text{O}_6$ , and  $\text{CoFe}_2\text{O}_4$ ) into elementary tetrahedrons is realized if the connoda  $\text{Ba}_2\text{Co}_2\text{Fe}_{16}\text{O}_{27} - \text{Fe}_2\text{Al}_2\text{O}_6$ , is available:

- 19)  $\text{Fe}_2\text{O}_3 - \text{BaFe}_{12}\text{O}_{19} - \text{Fe}_2\text{Al}_2\text{O}_6 - \text{BaCo}_2\text{Fe}_{16}\text{O}_{27}$ ;
- 20)  $\text{Fe}_2\text{O}_3 - \text{Fe}_2\text{Al}_2\text{O}_6 - \text{CoFe}_2\text{O}_4 - \text{BaCo}_2\text{Fe}_{16}\text{O}_{27}$ .

The analogous situation is peculiar for the tetrahedral domain circumscribed by the points of the compositions of compounds  $\text{BaFe}_{12}\text{O}_{19}$ ,  $\text{Fe}_2\text{Al}_2\text{O}_6$ ,  $\text{CoFe}_2\text{O}_4$ ,  $\text{Al}_2\text{O}_3$  that has the connoda  $\text{Fe}_2\text{Al}_2\text{O}_6 - \text{Ba}_2\text{Co}_2\text{Fe}_{16}\text{O}_{27}$ . The partition of this domain into elementary tetrahedrons is possible using the  $\text{Al}_2\text{O}_3 - \text{Ba}_2\text{Co}_2\text{Fe}_{16}\text{O}_{27}$  connoda:

- 21)  $\text{BaFe}_{12}\text{O}_{19} - \text{Fe}_2\text{Al}_2\text{O}_6 - \text{BaCo}_2\text{Fe}_{16}\text{O}_{27} - \text{Al}_2\text{O}_3$ ;
- 22)  $\text{Fe}_2\text{Al}_2\text{O}_6 - \text{BaCo}_2\text{Fe}_{16}\text{O}_{27} - \text{CoFe}_2\text{O}_4 - \text{Al}_2\text{O}_3$ .

The further tetrahedration requires the analysis of phase equilibriums for solid-phase exchange reactions:

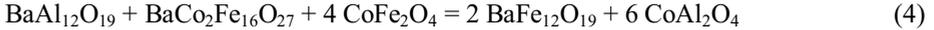


The computation data  $\Delta G = f(T)$  for the reactions (1)–(3) are given in the Table 2 and show that it is preferable for the reaction to progress in the direct direction except for the reaction (3) that is reversible at 1565 K, and in the high-temperature range the  $\text{BaAl}_2\text{O}_4$ , and  $\text{CoFe}_2\text{O}_4$  phase combination is more stable thermodynamically in comparison with  $\text{Ba}_2\text{Co}_2\text{Fe}_{16}\text{O}_{27}$  and 6  $\text{CoAl}_2\text{O}_4$ .

**Table 2.** Computation data  $\Delta G = f(T)$

Reaction No	Gibbs energy value, kJ, at T, K	
	1000	1500
1	-656	-1493
2	-340	-408
3	-232	-39
4	-888	-1532
5	-572	-447

The phase variability due to the reaction reversibility (3) can be of importance only for particular cases, because the reactions (1) and (2) are more advantageous thermodynamically. In the indicated thermodynamic range the changes in the signs  $\Delta G$  of summative reactions (4) = (1) + (3), (5) = (2) + (3), are not observed (Table 2):



A negative value of  $\Delta G$  for the reactions (4) and (5) is indicative of the coexistence of  $\text{BaAl}_{12}\text{O}_{19}$  with  $\text{CoAl}_2\text{O}_4$  and  $\text{BaCo}_2\text{Fe}_{16}\text{O}_{27}$  with  $\text{CoAl}_2\text{O}_4$ . The elementary tetrahedrons are closed by the connoda  $\text{BaAl}_{12}\text{O}_{19} - \text{CoAl}_2\text{O}_4$ , respectively.

- 23)  $\text{CoO} - \text{BaFe}_{12}\text{O}_{19} - \text{CoAl}_2\text{O}_4 - \text{BaAl}_2\text{O}_4$ ;
- 24)  $\text{BaFe}_{12}\text{O}_{19} - \text{CoAl}_2\text{O}_4 - \text{BaAl}_2\text{O}_4 - \text{BaAl}_{12}\text{O}_{19}$ ;
- 25)  $\text{BaFe}_{12}\text{O}_{19} - \text{CoAl}_2\text{O}_4 - \text{BaAl}_{12}\text{O}_{19} - \text{Al}_2\text{O}_3$ .

The connoda  $\text{BaCo}_2\text{Fe}_{16}\text{O}_{27} - \text{CoAl}_2\text{O}_4$  completes the partition of analyzed system into elementary tetrahedrons:

- 26)  $\text{CoO} - \text{BaFe}_{12}\text{O}_{19} - \text{BaCo}_2\text{Fe}_{16}\text{O}_{27} - \text{CoAl}_2\text{O}_4$ ;
- 27)  $\text{CoO} - \text{BaFe}_{12}\text{O}_{19} - \text{BaCo}_2\text{Fe}_{16}\text{O}_{27} - \text{Al}_2\text{O}_3$ ;
- 28)  $\text{CoO} - \text{BaCo}_2\text{Fe}_{16}\text{O}_{27} - \text{CoAl}_2\text{O}_4 - \text{CoFe}_2\text{O}_4$ ;
- 29)  $\text{BaCo}_2\text{Fe}_{16}\text{O}_{27} - \text{CoAl}_2\text{O}_4 - \text{CoFe}_2\text{O}_4 - \text{Al}_2\text{O}_3$ .

Therefore, the subsolidus structure of  $\text{BaO} - \text{CoO} - \text{Fe}_2\text{O}_3 - \text{Al}_2\text{O}_3$  system was defined only by 6 "internal" connodas that distinguish 29 elementary tetrahedrons that define all the 2, 3 and 4-phase combinations of coexistent compounds of the system (Fig.1).

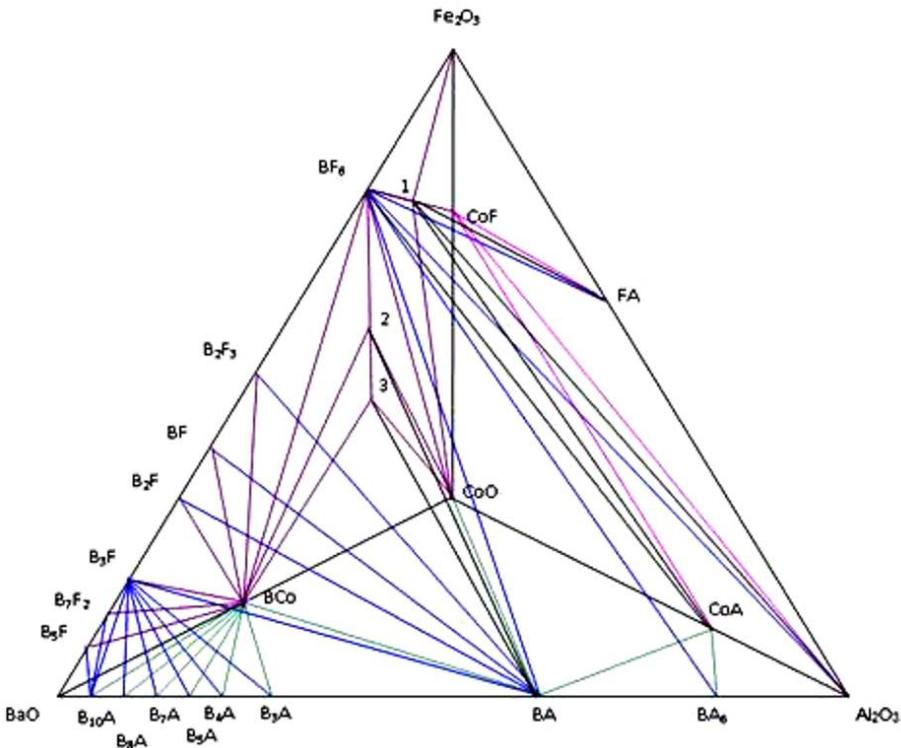


Fig. 1. Subsolidus structure of the  $\text{BaO} - \text{CoO} - \text{Fe}_2\text{O}_3 - \text{Al}_2\text{O}_3$  system.

## 5 Research-related conclusions, outlooks and further progress in this area

Theoretical research done for the subsolidus of  $\text{BaO} - \text{CoO} - \text{Fe}_2\text{O}_3 - \text{Al}_2\text{O}_3$  system is of interest for advanced applications of this system. For example, the obtained data can be used for the production of ferrimagnetic cementing materials using the principle of reaction-formed structures that have high values of the refractoriness combined with good protective properties to prevent the impact of electromagnetic radiation of different wave lengths. A promising field for the application of obtained data is the cement production technology. The produced cement can be used independently and as a binding agent to produce special concretes and materials that retain their properties under the action of high-frequency electromagnetic radiation.

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