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*S.M. Logvinkov ^a, N.S. Tsapko ^b, H.M. Shabanova ^c, O.M. Borysenko ^c, M.Yu. Ivashchenko ^a***ANALYSIS OF THERMODYNAMIC CONJUGATION OF SOLID-PHASE EXCHANGE REACTIONS IN THREE- AND FOUR-COMPONENT SYSTEMS**^a O.M. Beketov National University of Urban Economy in Kharkiv, Kharkiv, Ukraine^b Research Institution «Ukrainian Research Institute of Environmental Problems», Kharkiv, Ukraine^c National Technical University «Kharkiv Polytechnic Institute», Kharkiv, Ukraine

To develop new materials with predefined properties, it is essential to accurately predict their phase composition across the entire concentration range of the system. This requires a detailed analysis of the subsolidus structures of multicomponent systems. However, thermodynamic conjugation complicates this task, since solid-phase reactions may proceed in different directions, thereby influencing the coexistence of phases. This article proposes a method for analyzing thermodynamic conjugation that accounts for the most complex cases of reaction direction changes. The method simplifies the determination of phase compositions in multicomponent systems and improves the accuracy of predictions, which is particularly important in materials science. The thermodynamic combination of solid-phase exchange reactions of the «2=2» type into the «3=2» interaction mechanism, demonstrated using the CoO–BaO–Fe₂O₃ system, is frequently encountered in the technological analysis of multicomponent systems. During the tetrahedration of four-component systems, more complex types of conjugation may occur, such as «3=3» and «2=4». While the analysis in these cases is more labor-intensive, the methodology remains essentially the same.

Keywords: phase composition, three-component system, four-component system, triangulation, tetrahedration, thermodynamically conjugated reaction.

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Introduction

The physicochemical basis for the development of modern superalloys, oxide-anodized electrochemical coatings, ceramic-matrix composites, sialons, and functional oxide materials with specific electromagnetic properties lies in the phase diagrams of multicomponent systems. Each graphical element of a phase diagram represents a strictly defined physicochemical state of the system, which can be altered by varying external parameters (most often temperature and pressure). This makes it possible to predict ongoing processes and to control the achievement of the desired properties

of the materials under development [1–4].

As the number of components in a phase diagram increases, part of the information is inevitably lost due to the limitations of graphical representation. Diagrams with more than four components are represented as hypothetical figures in multidimensional space (pentatopes, hexatopes, and, in the general case, polytopes). They can be analyzed through the study of their subsystems using topological methods and matrix analysis [5,6]. However, even many three- and four-component phase diagrams remain underexplored. The lack of such data prevents the

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Analysis of thermodynamic conjugation of solid-phase exchange reactions in three- and four-component systems

adequate construction of more complex multicomponent diagrams based on them as subsystems. For these reasons, in materials science practice, three- and four-component phase diagrams, particularly those displayed in the regular concentration triangle or in the regular concentration tetrahedron, are the most widely used.

Methods for constructing and analyzing phase diagrams have been evolving since the inception of physicochemical analysis and have been summarized in numerous works by materials scientists [5,6]. The fundamental monograph [5] describes the application of thermodynamic analysis to establish the subsolidus structure of phase diagrams (i.e., below the melting temperatures of the various phases in the system). However, that monograph pays only limited attention to reversible solid-phase reactions and to solid-phase reactions capable of thermodynamic conjugation, which may alter the subsolidus structure of phase diagrams. In a later monograph [7], the main methods of thermodynamic analysis of multicomponent systems involving such types of chemical interactions are presented. More recently, new principles for analyzing these systems and methods for verifying constructions in their subsolidus regions have been developed [8–10].

The aim of this work is to present to specialists developing new materials the principles of thermodynamic analysis of three- and four-component systems under complex conditions, specifically, when thermodynamic conjugation of exchange-type solid-phase reactions may occur in their concentration regions.

Results and discussion

Theoretical background

The study of the subsolidus structure of three-component systems involves the establishment of all stable combinations of compounds of the system in combinations of two and three, and in four-component systems – also in four. In the composition of stable combinations of phases (the term «phase» is more correct, since the degree of stoichiometry of compounds is not always reliably known), they are usually called «coexistent», that is, chemically not interacting with each other.

The points of composition of coexisting phases are connected to each other by straight line segments that do not intersect on the phase diagrams and form elementary triangles in three-component systems, and, in four-component systems, also elementary tetrahedra. Triangulated and tetrahedral subsolidus regions of multicomponent systems make it possible to predict the phase composition of materials, which remains

unchanged within a certain temperature range. Using experimental methods, even the triangulation of three-component systems is an extremely labor-intensive procedure that requires planning experiments, preparing batches and samples, applying various heat treatment modes, and studying the microstructure and phase composition using modern instrumental methods of physicochemical analysis. The procedure can be simplified by thermodynamic analysis of the direction of solid-phase exchange reactions, which are modeled for all possible pairwise combinations of compounds in the systems [7]. The number of double, triple, and quaternary compounds (during tetrahedration) determines the number of possible model solid-phase reactions and, accordingly, the degree of complexity of the subsolidus structure of the system [11]. Methods of thermodynamic calculation and determination of equilibrium phase combinations from the initial reagents or from the products of solid-phase reactions, according to the calculated Gibbs energy changes (ΔG) at specific temperatures, are well known and described in detail [5,7]. Triangulation and tetrahedration of systems are based on the results of thermodynamic analysis and are subject to experimental verification only for a limited number of reference compositions.

A special situation with triangulation or tetrahedration arises in cases where the coexistence of phases is limited to a certain temperature, above which they enter into solid-phase chemical interaction. In this case, reactions of the type «2=2» and «2=3» (or vice versa) can occur in three-component systems, while reactions of the type «3=3» and «2=4» (or vice versa) may occur in four-component systems. Here, the equal sign separates the number of phases in the initial reagents and in the reaction products. The change in the combination of coexisting phases in reactions of the «2=2» type at a certain temperature actually corresponds to their reversibility. Graphically, such a situation corresponds to the disappearance of one connode and the appearance of another connode intersecting the first, which is referred to as the restructuring of connodes [7].

In other types of reactions, thermodynamic conjugation of two or three solid-phase reactions of a simpler type is realized. In essence, thermodynamic conjugation is analogous to the concept of a steady state in chemical kinetics. However, while the establishment of a steady state involves the comparison of reaction rates, thermodynamic conjugation involves the comparison of ΔG values. Conjugation of solid-phase reactions represents a kind of response of a physicochemical system to increasing temperature, and, in accordance with the Le Chatelier-Brown principle, other phase combinations with higher melting

temperatures are formed among the reaction products. In fact, the system acquires the ability to undergo structural–phase self-organization in order to preserve its integrity under increasing thermal loads [7].

Accordingly, the identification of conjugation processes provides specialists with a new toolkit for developing materials with tailored properties. At the same time, the complexity of the subsolidus structure of systems also increases, since different temperature ranges correspond to different variants of triangulation or tetrahedration. In addition, non-trivial situations arise in the thermodynamic analysis of coexisting phases during the conjugation of solid-phase reactions. The correctness of the triangulation is verified using Kurnakov's formula for the number of elementary triangles (t) [5]:

$$t = 1 + N_2 + 2N_3, \quad (1)$$

where N_2 and N_3 are the number of binary and ternary chemical compounds, respectively.

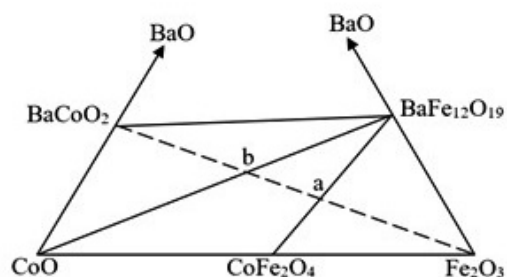
At the same time, the formula, also proposed by Kurnakov for checking the number of internal connodes (k), that is, those not belonging to the sides of the concentration triangle in three-component systems, has been undeservedly forgotten:

$$k = N_2 + 3N_3. \quad (2)$$

The correctness of the tetrahedration is not verified, which is due to the situation of the existence of an «empty contour» discussed in ref. [5].

Thermodynamic analysis for the systems under study

Complex aspects of thermodynamic analysis of the conjugation of solid-phase reactions are illustrated using the example of triangulation of the specific system $\text{CoO}–\text{BaO}–\text{Fe}_2\text{O}_3$ (the following abbreviations are adopted: «Co» – CoO , «B» – BaO , «F» – Fe_2O_3 , «BCo» – BaCoO_2 , « BF_6 » – $\text{BaFe}_{12}\text{O}_{19}$, and «CoF» – CoFe_2O_4). The analysis was carried out in a simplified form, without taking into account the existence of ternary chemical compounds. To establish the connodes in part of this system (Fig. 1), model solid-phase reactions were compiled, and calculations of their ΔG values were performed at temperatures in the range of 1000–1800 K (Table 1).



Scheme of the subsolidus structure of the system under study

The negative sign of ΔG for reaction No. 1 (Table 1) indicates the thermodynamic preference for its progression in the forward direction (from left to right). Therefore, the compounds in the reaction products (BCo and F) must coexist. A graphical representation of reaction No. 1 is the intersection of the hypothetical connodes: CoF and BF_6 with BCo and F (point «a», Fig. 1). The composition of the conditional compound at point «a» corresponds to the total amount of components on either the left or the right side of the equation for reaction No. 1, that is, CoBF_7 .

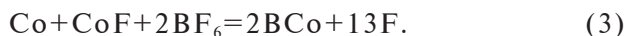
Note that reaction No. 2 (Table 1) has the same reaction products as reaction No. 1. However, the sign of ΔG for reaction No. 2 is positive, which indicates that the thermodynamically favorable direction is the reverse one (from right to left). Therefore, the compounds in the initial reactants must coexist. The graphical representation of reaction No. 2 (Table 1) is the intersection of the hypothetical tie lines: Co and BF_6 with BCo and F (point «b», Fig. 1). Accordingly, the composition of the conditional compound at point «b» is represented by the total quantity of components on either the left or the right side of the equation for reaction No. 2 – BCoF_6 .

Essentially, reactions No. 1 and 2 are competitive and share common chemical compounds in their interaction mechanisms. If the possibility of thermodynamic conjugation were not taken into account, one could conclude that there should be a connode between BCo and F, since the modulus of the ΔG values for reaction No. 2 is smaller than the corresponding modulus of the ΔG values for reaction

Table 1
Values of ΔG as a function of temperature for solid-phase exchange reactions in the $\text{CoO}–\text{BaO}–\text{Fe}_2\text{O}_3$ system

Reaction number	Reaction equation	ΔG , kJ/mol				
		1000 K	1200 K	1400 K	1600 K	1800 K
1	$\text{CoF} + \text{BF}_6 = \text{BCo} + 7\text{F}$	–2021	–2099	–2178	–2257	–2336
2	$\text{Co} + \text{BF}_6 = \text{BCo} + 6\text{F}$	463	556	683	846	1046

No. 1. However, solid-phase reactions No. 1 and 2 (Table 1), due to competition for common compounds, can be conjugated, combining into a new interaction mechanism of the «3=2» reaction type. In the simplest case, the equation of such a reaction is obtained by algebraic summation of reactions No. 1 and 2 (with the value of ΔG also determined by algebraic summation):



It is not difficult to calculate that the values of ΔG for reaction No. 3 remain negative (in particular, $\Delta G_{1000\text{ K}} = -1558$ and $\Delta G_{1800\text{ K}} = -1290$ kJ/mol), which confirms the previously assumed coexistence of BCo and F. However, reaction No. 3 is only one of the possible variants of thermodynamic conjugation of reactions No. 1 and 2 (Table 1), since the geometric representation in this case is the intersection of a hypothetical elementary triangle with vertices at the compositions of Co, CoF, and BF_6 , and a hypothetical connode between the compositions of BCo and F according to the mechanism of reaction No. 3 (Fig.).

Therefore, the set of possible reactions for different variants of the conjugation of reactions No. 1 and 2 (Table 1) will correspond to the compositions between points «a» and «b» in Fig. Accordingly, the solution for the conjugation variant of reaction No. 3 corresponds to the hypothetical composition $\text{B}_2\text{Co}_2\text{F}_{13}$ (or $\text{BCoF}_{6.5}$), which illustrates that the point lies in the middle of segment «ab» (Fig.), since point «a» corresponds to the composition CoBF_7 and point «b» to CoBF_6 .

For the thermodynamic analysis to be complete, it is necessary to show that none of the solutions in the variants of conjugation of reactions No. 1 and 2 can yield a positive value of ΔG , which would exclude the presence of a connode between BCo and F. The thermodynamic conjugation variants of interest are obtained by multiplying the masses of all compounds in one of the reactions by the same factor. According to the corollary of the Gibbs phase rule [6], thermodynamic equilibrium does not depend on the proportional change in the masses of coexisting phases. In particular, if we take a factor of five for reaction No. 2 and perform an algebraic summation with reaction No. 1, we obtain the conjugation variant:



In reaction No. 4, compared to reaction No. 3, only the stoichiometric coefficients changed, while the combinations of compounds in the initial reactants

and in the reaction products remained the same. Accordingly, by multiplying the value of ΔG for reaction No. 2 (such factors in the theory of chemical reaction kinetics are called run coefficients) and adding it to the values of ΔG for reaction No. 1 (Table 1), it is easy to calculate the values of ΔG for reaction No. 4 and verify that they are positive over the entire temperature range: $\Delta G_{1000\text{ K}} = 294$ and $\Delta G_{1800\text{ K}} = 2894$ kJ/mol.

In this case, the graphical representation of reaction No. 4 corresponds to the composition point of the hypothetical compound $\text{B}_6\text{Co}_6\text{F}_{37}$ (or $\text{BCoF}_{6.16(6)}$), which is shifted closer to point «b» along segment «ab» in Fig. Accordingly, the ternary combination of phases in the elementary triangle Co–CoF– BF_6 is thermodynamically more favourable, while BCo and F do not coexist (Fig.). The connode BCo–F is interrupted, does not maintain its integrity, and therefore cannot fully intersect the triangle Co–CoF– BF_6 . The «interrupted» option is rejected, and the «complete» one is accepted.

In essence, the stability of the CoF– BF_6 junction is maintained by thermodynamic conjugation and the additional presence of Co in the three-phase combination. Thermodynamic conjugation of solid-phase exchange reactions of the «2=2» type into an interaction mechanism of the «3=2» type is often encountered in the technological practice of analyzing multicomponent systems. The situation considered for the CoO–BaO– Fe_2O_3 system can be generalized in the form of principles:

1) If for both conjugated reactions of the type «2=2» $\Delta G < 0$, then the two-phase combination of chemical compounds in the reaction products of the type «3=2» is stable; accordingly, in the subsolidus structure of the system, there is a connode between the points of the compositions of these compounds.

2) If for both conjugated reactions of the type «2=2» $\Delta G > 0$, then the three-phase combination of chemical compounds in the initial reactants of the reaction of the type «3=2» is stable; accordingly, in the subsolidus structure, there is an elementary triangle, the vertices of which correspond to the points of the compositions of these compounds.

3) If for conjugated reactions of the type «2=2» the values of ΔG have different signs, then additional analysis is required using the above method.

During tetrahedration of four-component systems, more complex types of conjugation may occur: «3=3» and «2=4». The analysis method in these cases is more labor-intensive but does not differ fundamentally from that discussed above, since the visual representation corresponds to the intersection of two triangles or a straight-line segment with a

tetrahedron, respectively. It is evident that the search for solution options should be carried out along the straight-line segment common to the intersecting triangles (reaction type «3=3») or along the straight-line segment bounded by the external elements of the tetrahedron structure (reaction type «2=4»).

Conclusions

Thermodynamic conjugation of solid-phase exchange reactions in three- and four-component systems is manifested in the ability to counteract external energy factors most effectively by changing the phase composition and corresponding microstructure while maintaining the integrity of the material. To obtain new materials with a set of predetermined properties, it is necessary to accurately predict their phase composition in any concentration region of the system, which becomes possible when analyzing the subsolidus structure of multicomponent systems. Thermodynamic conjugation of solid-phase reactions complicates the subsolidus structure, and new methods are required to correctly determine all possible coexisting phases for various combinations of compounds in the system. The method of analyzing thermodynamic conjugation considered here takes into account the most non-trivial case, in which solid-phase reactions proceed in different directions. The principles for establishing coexisting phases during thermodynamic conjugation allow materials scientists to simplify the analysis of the subsolidus structure of multicomponent systems and to increase the accuracy of predicting the phase composition in any concentration range.

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АНАЛІЗ ТЕРМОДИНАМІЧНОЇ СПРЯЖЕНОСТІ ТВЕРДОФАЗНИХ РЕАКЦІЙ ОБМІНУ У ТРИ- ТА ЧОТИРИКОМПОНЕНТНИХ СИСТЕМАХ

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Для розробки нових матеріалів із заданими властивостями важливо точно прогнозувати їх фазовий склад у всій концентраційній області системи. Це потребує детального аналізу субсолідусної будови багатокомпонентних систем. Однак термодинамічне спряження ускладнює цей аналіз, оскільки твердофазні реакції можуть протікати в різних напрямках, впливаючи на співіснуючі фази. У цій статті запропоновано методику аналізу термодинамічної спряженості, що враховує найбільш складні випадки зміни напрямку реакцій. Це дозволяє спростити визначення фазового складу багатокомпонентних систем та підвищити точність його прогнозування, що особливо важливо для матеріалознавства. Термодинамічне поєднання твердофазних реакцій обміну типу «2=2» в механізм взаємодії типу «3=2», яке розглянуто на прикладі системи CoO–BaO–Fe₂O₃, часто зустрічається в технологічній практиці аналізу багатокомпонентних систем. Під час тетраедрації чотирикомпонентних систем зустрічаються ситуації складніших типів спряження: «3=3» та «2=4». Методика аналізу у цих випадках більш трудомістка, але не відрізняється від розглянутої.

Ключові слова: фазовий склад, трикомпонентна система, чотирикомпонентна система, триангуляція, тетраедрація, термодинамічне спряження реакцій.

ANALYSIS OF THERMODYNAMIC CONJUGATION OF SOLID-PHASE EXCHANGE REACTIONS IN THREE- AND FOUR-COMPONENT SYSTEMS

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To develop new materials with predefined properties, it is essential to accurately predict their phase composition across the entire concentration range of the system. This requires a detailed analysis of the subsolidus structures of multicomponent systems. However, thermodynamic conjugation complicates this task, since solid-phase reactions may proceed in different directions, thereby influencing the coexistence of phases. This article proposes a method for analyzing thermodynamic conjugation that accounts for the most complex cases of reaction direction changes. The method simplifies the determination of phase compositions in multicomponent systems and improves the accuracy of predictions, which is particularly important in materials science. The thermodynamic combination of solid-phase exchange reactions of the «2=2» type into the «3=2» interaction mechanism, demonstrated using the CoO–BaO–Fe₂O₃ system, is frequently encountered in the technological analysis of multicomponent systems. During the tetrahedration of four-component systems, more complex types of conjugation may occur, such as «3=3» and «2=4». While the analysis in these cases is more labor-intensive, the methodology remains essentially the same.

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