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Compositional behavior of heat capacity and magnetic properties in double spinel ferrites

Machaladze Tengiz, Khundadze Mzia *, Varazashvili Vera, Tsarakhov Murman, Rokva Lela and Jorbenadze Rusudan

N. Landia Laboratory of Thermochemistry, Institute of Inorganic Chemistry and Electrochemistry, Iv. Javakhishvili Tbilisi State University, Tbilisi, Georgia.

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Abstract

The purpose of the work is to identify the effects of the substitution of paramagnetic ions in ferromagnetic ferrites by diamagnetic components of zinc and aluminum. The following isomorphic spinel series are the object of review: $Me_{(1-x)}Zn_xFe_2O_4$ (Me – Ni, Co, Li, Cu, Mg) and $Li_{0.5}Fe_{2.5-x}Al_xO_4$ (x= 0-2.5). The single-phased samples were synthesized by ceramic procedure. Heat capacity near standard temperature C_p (298.15) was studied using calorimeter methods: drop calorimeter and DSC. Magnetic parameters – magnetic moments (M) and Curie temperatures (T_c) - were determined. All Me-Zn ferrite systems are characterized by dome-shaped correlations between heat capacity C_p (298) and composition (x), which have maximum values in the equimolecular region (x=0.4-0.6). This fact coincides with the nature of the compositional dependence of magnetic moments and indicates a significant effect of the excess magnetic contribution on the total heat capacity. A noticeable influence of the cooperative Jahn-Teller effect and the associated tetragonal distortion of the cubic lattice of Cu-Zn ferrites with a low Zn content (x<0.2) is observed, which causes overestimated values of the heat capacity of these compositions. Curie temperatures reveal the rectilinear decrease with increasing diamagnetic Zn and Al content in ferrite systems. The results are presented by graphs and tables.

Keywords: Double ferrite systems; Heat capacity; Calorimetry; Magnetic properties

1. Introduction

Currently, new compositions based on mixed inorganic oxides - ferrites are intensively developed. The interest in ferrite compositions has grown especially with the development of nanotechnology, which is widely reflected in most new publications on different aspects of ferrites [1-6]. The extraordinary variety of possible compositional combinations, as well as their new and often unique properties useful for practical applications, leads to a comprehensive study of multicomponent oxides in many directions, including the study and reviewing of their thermodynamic characteristics. Even though today a large amount of data on the thermal parameters of oxide compounds are accumulated in many reference books, computer databanks, and numerous publications, however, the existing experimental research methods are not yet able to cover the growing demand for the characteristics of new oxide developments in a short time. Meanwhile, these data are necessary for a successful solution of several practical problems, such as targeted selection of promising compositions and preliminary assessment of the properties of new developments; search for the optimal energy regime for the synthesis of multicomponent oxides; problems associated with their thermal and compositional stability; the choice of suitable conditions for their long-term operation.

Along with experimental ways, various empirical and semi-empirical calculation methods for estimating the thermal properties of oxide compounds are increasingly being used [7-10]. In this regard, it seems very promising to study and

^{*} Corresponding author: Khundadze Mzia

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identify the relationships between composition, physical properties (magnetic, electrical, thermal, mechanical, etc.), and characteristics of constituent components (ions). The correlations revealed for certain groups and classes of compounds will open up the possibility for a preliminary assessment and prediction of similar properties for compositions within isostructural families of compounds, avoiding complex experimental studies. This, in turn, will contribute to the development of new calculation methods, and intensification of thermodynamic estimates.

The purpose of this work is to identify the dependences of thermal and magnetic properties on compositional variables for several complex oxide systems of spinel group and to present the corresponding correlations suitable for practical use.

In the ternary oxide system Me'O-Me"O-Fe₂O₃, several compounds and solid solutions are formed that belong to different crystal groups. Among them, the compounds with the general formula Me'(1-x)Me"(x) Fe2O4 (where Me 'and Me" are metal ions), having a spinel-type cubic crystal structure, are of special interest. Spinel ferrites are widely used in various fields of modern technology as ferromagnetic semiconductors, catalysts, elements of electronic equipment, etc. [5, 6]. Using cationic substitution, purposeful searches for new combined compositions are constantly being carried out and the areas of their practical application are being identified. It is obvious that the identification of general trends in the influence of such factors as temperature, composition, mass and volume of ions, crystal and magnetic structure, on thermodynamic properties, will promote a favorable basis for obtaining reliable results for accelerating the development of new compositions with advanced and predicted properties and for working out the corresponding technologies.

In the present work, research is underway to identify the effect of magnetic dilution on the various properties of solid solutions of the following double ferrite-spinel systems: Ni(1-x)Zn(x)Fe2O4; Co(1-x)Zn(x)Fe2O4; Li(x)Zn(2.5-x)Fe2O4; Cu(1-x)Zn(x)Fe2O4; Mg(1-x)Zn(x)Fe2O4, as well as ferrite-aluminates with the general formula Li0.5Fe(2.5-x)Al(x)O4 (x = 0-2.5).

The data on the structural and magnetic characteristics of these spinel-ferrites are presented in numerous publications and mainly collected in [5, 6]. On the base of Neel's two sub-lattice model the principal tendency of cations for distribution along the A - tetrahedral and B - octahedral sites of spinel-like lattice are explained. It has been shown that the cationic composition and the way of their distribution over structural sites is a determining factor for their magnetic properties, and also there is extensive information about practically important magnetic and electrical properties. Thermochemical properties are also presented in several publications, including the previous works of the authors of this article [7, 8, 12, and 13].

Based on a joint analysis of literature data and the results of our experiments, the following interdependencies for ferrite systems are presented and discussed: the nature of the change in heat capacity depending on the cationic composition and magnetic properties; the influence of the type of distribution of cations over the crystallographic sites of the spinel structure on their properties; relationships between composition and magnetic parameters (Curie temperature -Tc, magnetic moment -M); influence of mass and size factors of cations; the contribution of phase transformations and anomalies (such as ferromagnetic transition, order-disorder transformation, Schottky anomaly and the Jahn-Teller effect) to the heat capacity. The heat capacity is chosen for reviewing, as the most structurally sensitive and the principal thermodynamic parameter used at the corresponding thermochemical calculations of technological processes, including determining Gibbs energy of formation.

2. Material and methods

The following data were used as initial indicators:

- Heat capacity data at standard (25°C) temperature Cp(298.15) obtained using the following calorimetry methods: 1. differentiation of high-temperature (25-1000°C) enthalpy functions H(T) determined using a drop adiabatic calorimeter [7]; 2. direct measurements of heat capacity carried out on a differential scanning calorimeter (DSC) [12, 13]. The accuracy of the data is within ± 2.0 and 1.5 %, respectively and they are in good agreement with each other.
- Characteristics of ferromagnetic properties: magnetic moments (M) near standard temperature measured by Sexsmith's weight dynamic method [7]; Curie temperatures (Tc) measured by the ballistic method [7] and by thermal analysis and DSC. Literature data [5, 6] were also applied.
- Phase transformations are identified and characterized by using calorimeters: HT-1500 ("Setaram" production), adiabatic drop calorimeter, DSC, and thermo-gravimetric studies [12, 13].

• The samples of ferrites were obtained by high-temperature processing of the starting oxides using ceramic technology. The compositional, structural, and magnetic characteristics of single-phased products meet the requirements and are presented in Table 1.

3. Result and Discussion

The investigated spinel ferrites and their solid solutions, in terms of cation composition and their distribution over octahedral [B] and tetrahedral (A) positions, are conditionally divided into the following subgroups: a) normal spinel (Me2+)[Me3+2]O4; b) reversed-[Me2+](Me3+2)O4 and c) intermediate - (Me2+x)Me3+1-x)[Me2+1-xMe3+1+x]O4, in which the position of cations depends on the composition, temperature and history of synthesis. Such a division in most cases is conditional and depends on the energy of preference of cations to that or another sublattice.

This paper presents binary ferrite systems that form the solid solutions, where the ferromagnetic component is gradually replaced by a diamagnetic one: a) solid solutions of normal and reversed spinel, where the diamagnetic component is normally structured zinc ferrite ZnFe2O4 (ZnF); b) some lithium ferrite-aluminates, where ferromagnetic iron is replaced by diamagnetic aluminum. The choice of these systems for study is because they are a series of solid solutions of ferromagnetic and diamagnetic components that will allow tracing the nature of the effect of such type of substitution on the thermo-physical and thermo-chemical properties discussed.





According to the cationic composition, the presented solid solutions of ferrites can be attributed to the following groups: spinel ferrites containing divalent ions – Me(1-x)Zn(x)Fe2O4 where Me=Co, Ni, Cu, Mg; ferrites that contain monovalent lithium ions Li0.5(1-0.5x)Zn(x)Fe(2.5-0.5x)O4; and lithium ferrite-aluminates Li0.5Fe(2.5-x)Al(x)O4 (x=0 - 2.5), where trivalent iron ions are replaced by aluminum ions. Despite belonging to the common crystalline group of spinel, a certain variety of physical properties (magnetic, electrical, etc.) of these ferrites is observed, which should affect the thermochemical parameters, in particular, the heat capacity.

3.1. Heat Capacity

The function of heat capacity of solids, including ferrites, in a general form can be represented as a complex value:

$$C_p = C(reg) + C(ex)$$
(1)

In addition to the main regular part C(reg) = C(L) + C(d) (where C(L) is the lattice-vibration heat capacity, C(d) is the difference between isobaric and isochoric heat capacities), equation (1) may contain excess C(ex) components, due to the effects of phase transformations, as well as some other anomalies, such as cationic and magnetic disordering processes, Schottky or Jahn-Teller effects, and some others. In the case of ferrimagnetic ferrites, the main part of C(ex) is the magnetic anomaly - Cm, associated with the energy required to reduce the degree of existing magnetic order.

In the present work the features of the change in heat capacity and magnetic characteristics depending on composition, dimensional factors (ionic radii, lattice parameter), and the effects of phase transformations and anomalies are reviewed.

3.1.1. Compositional effects in double ferrites:

With gradual dilution of ferrimagnetic ferrites of nickel, cobalt, copper, magnesium and lithium with diamagnetic zinc ferrite, a gradual rectilinear increase in the lattice parameter (a) and a decrease in the Curie temperature - Tc are observed (Fig.1, Table 1). However, the saturation magnetic moments curves, Ms(x), pass through a maximum at intermediate compositions of x=0.4-0.6 (Figures 2-7) that is connected with a special behavior of the energy of stabilization at redistribution of cations over tetrahedral and octahedral sites of spinel structure while gradual adding zinc ions [5].

As an example, in Fig. 1 shows the relationship between Curie temperature (Tc) and lattice parameter (a) for a lithiumzinc ferrite system. A similar behavior is also observed in the case of other Me-Zn ferrite systems (Table 1). With an increase in the content of the diamagnetic component, a linear decrease in Tc is observed in all systems. This is due to such a redistribution of cations over the lattice sites, which causes a change in the values of the exchange integrals that determine the Curie temperature [5]. The influence of the size factor may also have some additional effect since the strength of magnetic interactions decreases due to an increase in the distance between the paramagnetic cations in the spinel lattice. As for the lithium iron-aluminate system, though the Curie temperature also decreases towards higher aluminum content, there are no clear signs of the influence of the ions size factor.

To identify the relationship between magnetic properties and heat capacity in a series of spinel solid solutions, first of all, consider the concentration dependences of the magnetic moment (M) and heat capacity near the standard temperature - Cp (298.15). Figures 2-7 illustrate the double systems of Me-Zn ferrites. As it is given in [5] and confirmed by our data (Table 1), with an increase in the concentration of diamagnetic zinc ion, the magnetic moment-composition curves take on a domed shape with a maximum near an equimolecular composition $x \approx 0.4$ -0.6. The curves of the dependence of heat capacity on composition have a similar form. The positions of the maximums on the heat capacity and magnetic moment curves coincide with each other within the error of the experimental data, which indicates a significant role of the magnetic contribution Cm to the total heat capacity.

All binary systems containing zinc (NiZnF, CoZnF, LiZnF, CuZnF, MgZnF) at standard temperature, in general, have the identical form of the concentration dependence of heat capacity Cp (as well as of magnetic saturation moments M). However, one should dwell on the following points: a) in the system of lithium-zinc ferrites, there is some difference in the position of peaks on the concentration curves of heat capacity (\sim 0.35) and magnetic moment (\sim 0.45). This may be due to the characteristic of lithium ferrite residual reflection of superstructural contribution to the heat capacity, which tends to sharply decrease as dilution by zinc ferrite [5]. b) Unlike the ferrites discussed above, copper and magnesium ferrites are very weak ferromagnets (Table 1). The magnetic moments and Curie temperatures of these ferrites, and solid solutions with zinc ferrite, are essentially determined by the degree of inversion of their spinel structure, the distribution of cations over the A and B interstices, and, consequently, by the conditions of synthesis and the way of thermal treatment.

Figure 4 shows the graphs of the concentration dependence of heat capacity Cp(298.15) and the magnetic moments (M) of the synthesized by us Cu-Zn ferrite samples. The general form of these curves is identical to the systems considered earlier. However, in the Cu-Zn system, the heat capacity of pure copper ferrite (x = 0) stands out sharply and has an overestimated value compared to the subsequent composition. This is due to the influence of the Jahn-Teller effect [5] characteristic of copper ions located both in octahedral and tetrahedral positions. It is known that in the presence of a sufficient amount of Jahn-Teller copper ions, cooperative ordering of tetragonally deformed polyhedrons occurs, and the cubic spinel structure transforms into a tetragonal structure with c/a>1. As a result, with a change in the structure, besides the lattice component C(reg), the excess part – C(ex) also would be changed, since in addition to the ferromagnetic contribution - Cm, another C(JT) component appears, due to the Jahn-Teller effect. With a small decrease in the content of copper ions, already at x<0.2, the bonds between locally deformed polyhedrons are permanently destroyed. Consequently, the cooperative nature of the Jahn-Teller effect and the tetragonal macroscopic deformation of the lattice disappear. As a result, the system returns to the spinel-type cubic structure. However, in the region x>0.2 local distortions of copper ions remain (the dynamic Jahn-Teller effect), which is reflected in the concentration curve of heat capacity [13].

A special picture exists in the case of solid solutions of lithium ferrite-aluminates (Fig. 7) of the Li0.5Fe2.5O4-Li0.5Al2.5O4 system. It is known that solid solutions in this system are formed only for compositions with the general formula Li0.5Fe(2.5-x)Al(x)O4, where x=0 - 2.5 [14]. Curve 2 in Fig.7 represents the compositional dependence of the magnetic moment - M (Table 1), which is built on the base of combined literary (up to x=0.7) [14, 15] and our data (x>0.7). In contrast to the systems considered earlier, with an increase in the mole fraction of lithium aluminate, the magnetic moment curve sharply decreases, reaching a minimum at x=0.7 and maintaining minimum values up to M=0

at x=2.5. The resulting picture is due to the complexity of the magnetic structure of the representatives of this system. In compositions with x>0.4, aluminum ions change their preference to sub-lattices and gradually redistribute from octahedral to tetrahedral positions. As a result, compositions are formed that are characterized by a compensation point for the magnetic moments of sub-lattices [14, 15], which leads to a sharp decrease in their magnetic moments. The peculiarity of the magnetic characteristics of ferrite-aluminates is reflected in the heat capacity function. On the heat capacity-composition curve of this system, there is a drop in the value of heat capacity in the region near x<0.7 (dashed line on curve 1), which correlates with a sharp decrease in the magnetic moment in the same region (Curve 2). Above x=0.7, the general view of the magnetic moment and heat capacity lines are similar.

A joint analysis of the obtained data shows that the concentration dependences of heat capacity of most solid solutions of binary ferrite systems have the same form and reveal a maximum at compositions close to equimolecular, which is consistent with the type of change in the magnetic moment. Based on this, it can be assumed that such a correlation reflects the effect of a significant ferromagnetic contribution to the heat capacity - Cm. However, at present it is difficult to estimate the value of Cm and separate this excess part from the total heat capacity since there are no data or a reliable calculation method for evaluation of its main regular component – C(reg).

Due to the lack of data for mixed oxide systems, solid solutions of metals and isomorphic double salt systems can be cited for comparison. It is known that continuous series of metallic solid solutions are characterized by extremums and peaks on the concentration dependence of some physical properties. For example, the microhardness of most solid solutions of binary metals has a minimum value at equimolecular compositions, which corresponds to the maximum binding energy and minimum heat capacity. Therefore, the presence of a maximum on the heat capacity curves of ferrite systems instead of the expected minimum can be associated with a noticeable effect of the ferromagnetic component on the total heat capacity of this class of compounds.

Composition of double ferrites	х	lattice parameter, a, (Aº)	Curie temperature, Tc (°C)	Magnetic moment Ms (σ _b)	Cp at 25°C J/K. mole	
Ni1-xZnxFe2O4	0.0	8.340	556	2.29	145.65	
	0.2	8.358	456	3.70	151.7	
	0.4	8.383	350	5.00	155.5	
	0.6	8.400	185	5.00	151.5	
	0.8	8.417	50	3.10	146.8	
	1.0	8.434	-	0.00	138.0	
Co _(1-x) Zn _x Fe ₂ O ₄	0.0	8.376	500	3.30	144.2	
	0.2	8.364	396	4.50	151.2	
	0.4	8.401	273	5.83	155.4	
	0.5	8.402	150	6.17	154.0	
	0.6	8.414	83[7]	5.90	151.2	
	0.8	8.425	50[7]	4.10	144.2	
	1.0	8. 433	-	0.00	137.5	
Li0.5(1-x)Zn(x)Fe(2.5-0.5x)O4	0.0	8.330	630	2.47	152.0	
	0.027	8.332	625	2.90	152.5	
	0.097	8.340	569	3.40	153.8	
	0.200	8.350	500	3.60	155.2	
	0.368	8.366	385	4.50	155.1	

Table 1. Characteristics of double ferrite systems

	0.500	8.380	300	4.00	152.6	
	0.692	8.400	140	2.00	145.4 139.7 138.0`	
	0.900	8.420	-	-		
	1.000	8.434	-	-		
Cu _(1-x) Zn _x Fe ₂ O ₄ (Cp* at 320K)	0.0	8.254 (tetra. c/a=1.06)	488	1.8	152.2	158.4*
	0.2	8.384	387	2.3	151.4	156.6
	0.4	8.396	237	2.3	152.9	160.9
	0.6	8.415	-	1.9	154.9	161.2
	0.8	8.432	-	1.3	151.0	155.5
	1.0	8.433	-	0.0	138.0	141.0
Mg _(1-x) Zn _x Fe ₂ O ₄	0.0	8.385	422	0.8	148.4	154.5
	0.2	8.389	312	1.9	150.3	157.2
	0.4	8.409	-	1.7	151.0	155.6
	0.6	8.410	-	0.9	149.6	153.5
	0.8	8.420	-	0.1	145.0	148.6
	1.0	8.435	-	0.0	138.0	141.1
Li _{0.5} Fe _{2.5-x} Al _x O ₄ (x=0-2.5).	0.0	8.35	630	2.60	152.6	
	0.5	8.26	476	0.33	140.9	
	1.0	8.15	367	0.16	136.0	
	1.5	8.13	96	0.14	129.8	
	2.0	7.99	-	-	121.6	
	2.5	7.90	-	-	110.8	

4. Conclusion

Double ferrite systems form some isomorphic solid solutions having spinel structures. The dominant factor in the physical-chemical and magnetic properties of these compounds is the ratio and properties of structure-forming cations. In this paper, the object of study is the effect of magnetic dissolution on the thermochemical and magnetic properties in double ferrite systems Me(1-x)Zn(x)Fe2O4 (Me - Ni, Co, Li, Cu, Mg) and Li0.5Fe2.5-xAlxO4 (x = 0-2.5). A gradual increase in the content of diamagnetic zinc in ferrites, and aluminum in ferrite-aluminates, leads to the formation of dome-shaped compositional dependences of the heat capacity Cp at standard temperature, which have maximal values at compositions close to equimolecular x=0.4-0.6. This fact coincides with the compositional behavior of the magnetic moments in the same series and confirms the significant influence of the excess magnetic component on the values of the total heat capacity. Dilution of ferromagnetic ferrites with diamagnetic zinc ferrite and lithium aluminate causes a linear decrease in the Curie temperature - Tc in the corresponding binary systems. A noticeable influence of the cooperative Jahn-Teller effect and the associated tetragonal distortion of the cubic lattice of Cu-Zn ferrites with a high copper content (x<0.2) is observed, which causes overestimated values of the heat capacity of this composition.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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