Crystal Growth, Magnetic Properties and Analysis of Possible Magnetic Ordering of $Ni_5Sn(BO_5)_2$ with Ludwigite Structure

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The ludwigite $Ni_5Sn(BO_5)_2$ belongs to a family of oxyborates, and has an ordered distribution of nonmagnetic Sn ions. This material is studied here using magnetic measurements. $Ni_5Sn(BO_5)_2$ does not show full long-range magnetic order, but exhibits a partial ordering at 73 K. A theoretical group analysis is performed and different models of the magnetic structure of $Ni_5Sn(BO_5)_2$ are discussed.

1. Introduction

Oxyborates represent a wide group of compounds, and these have been studied for a considerable time.[1–5] Although oxyborates grow in different structures, almost all of these structures include low-dimensional elements such as walls,[1] ladders,[2] chains,[3] and ribbons.[4] The physical properties of oxyborates are interesting and vary widely due to the geometrical features of their structures, including charge ordering,[1] noncollinear magnetism,[2] and spin-glass states.[5]

The ludwigites are one of the most interesting groups of oxyborates. Compounds with a ludwigite structure have been widely studied, although it is still unclear which interactions are important in the formation of magnetic ordering in many compounds.[5] For example, the magnetic properties change dramatically when Sn is substituted for Ti in $Co_5Ti(BO_5)_2$.[6,7] The nonmagnetic ions of both Ti and Sn occupy the same position in the crystal structure, and the difference in the cell parameters is less than 2%, and it could therefore be expected that the magnetic properties would be similar, arising from the magnetic Co ions; however, in $Co_5Ti(BO_5)_2$, the magnetic transition to a spin glass state occurs at 20 K, whereas in $Co_5Sn(BO_5)_2$ long-range magnetic ordering occurs at a higher temperature of 84 K, as for the other Co-based ludwigites.

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It is possible that certain other interactions play a main role in magnetic ordering, rather than the superexchange Co–O–Co interactions. It is also possible that other non-superexchange interactions play a primary role in the formation of the magnetic order in $Co_5Ti(BO_5)_2$ and $Co_5Sn(BO_5)_2$, since the superexchange interactions are very close and the structure of the connections is the same.

There is a similar set of Ni-based ludwigites with the formula $Ni_5Me(BO_5)_2$, in which Me = Ti, Ge,Sn,Zr. Although all of these compounds were grown by Bluhm[8–10] and Stenger,[11] only the crystal structure was studied. The ludwigite $Ni_5Sn(BO_5)_2$ does not belong to this set, since its nonmagnetic ions (Sn) are ordered.[10] This is interesting, since there is no link between nonmagnetic ion ordering and ionic radius. The ludwigites $Ni_5Ge(BO_5)_2$, $Ni_5Zr(BO_5)_2$, and $Ni_5Ti(BO_5)_2$ have a disordered distribution of nonmagnetic ions, with smaller (for Ge) or larger (for Ti and Zr) radii than the Sn ion. An interesting question is how the ordered distribution of Sn influences the magnetic properties of $Ni_5Sn(BO_5)_2$, and whether the unusual properties of $Ni_5Ge(BO_5)_2$ arise.[12] In a previous study, we reported the very interesting magnetic properties of $Ni_5Ge(BO_5)_2$. An exchange bias in the hysteresis loop and strong anisotropy in the paramagnetic region were found, which is unusual for Ni^{2+} in octahedral surroundings. In this paper, we report the crystal growth, theoretical group analysis, magnetic properties, and an analysis of the possible magnetic structure.

2. Crystal Growth

Single crystals of $Ni_5Sn(BO_5)_2$ oxyborate were synthesised using the flux technique. A stoichiometric ratio of the crystal-forming oxides $5NiO{:}SnO_2{:}B_2O_3$ was dissolved in a mixture of $Bi_2Mo_3O_{12}{:}2.4B_2O_3{:}2Li_2O$ oxides. The concentration of the crystal-forming oxides was 7% (weight percentage). The flux was prepared by the sequential melting of the flux components in a platinum crucible at a temperature of $T = 1100\,^{\circ}C$: initially, the mixture of Bi_2O_3–MoO_3–B_2O_3 was melted, and the Li_2CO_3 carbonate added gradually; following this, for better solubility, NiO oxide was added gradually, and the SnO_2 oxide was then added. The weight of the flux was 91.6 g.

Small single crystals of $Ni_5Sn(BO_5)_2$ oxyborate were grown using a spontaneous nucleation process. After the flux had been prepared and homogenized for 2 h at a temperature of $T = 1100\,^{\circ}C$, the temperature in the furnace was rapidly reduced (at a rate of cooling of $dT/dt = 100\,^{\circ}C$/day) to $T = 980\,^{\circ}C$, and then more slowly, at a rate of $dT/dt = 16\,^{\circ}C$/day. Twenty-four hours later, the crucible was extracted from the furnace, and the flux was poured out. Single crystals in the form of tiny, elongated green prisms were formed, with a size of about $0.1 \times 0.2 \times 1$ mm, and these were separated using a 20% aqueous solution of nitric acid.

3. Structure

Powder X-ray diffraction of $Ni_5Sn(BO_5)_2$ showed that the structure of our crystal was the same as the structure solved by Bluhm in ref. [10]. $Ni_5Sn(BO_5)_2$ belongs to the *Pnma* (No. 62) space group, with lattice parameters $a = 9.301$ Å, $b = 12.275$ Å, $c = 6.102$ Å. Sn ions are ordered, and occupy the 4c position (**Figure 1**).

In general, the structure of ludwigites contains two subsystems, which are three-leg ladders formed by ions in positions 4-2-4 and 3-1-3. The passes of exchange interactions between subsystems form triangles. These geometrical features of the structure are expected to lead to frustrations. The noncollinear ordering in Fe_3BO_5[2] and Cu_2MnBO_5[13] appears due to the frustration of the exchange interactions between subsystems. In the case of $Ni_5Sn(BO_5)_2$, one of the subsystems contains nonmagnetic ions of Sn. An ordered distribution of Sn ions should either favour a magnetic ordering or destroy it.

4. Magnetic Measurements

Magnetic measurements of $Ni_5Sn(BO_5)_2$ were performed using PPMS-9. Due to the small size of the crystals ($0.1 \times 0.2 \times 1$ mm), it was impossible to carry out measurements on only one crystal, and all measurements were therefore conducted on polycrystalline samples. The temperature dependencies of magnetization shown in **Figure 2**a were obtained at 2–300 K, with an applied field $H = 1$ kOe in two regimes: zero-field cooled (ZFC) and field cooled (FC). The magnetization curves show a sharp kink, indicating the occurrence of magnetic ordering near 73 K. The curves corresponding to FC and ZFC regimes show splitting.

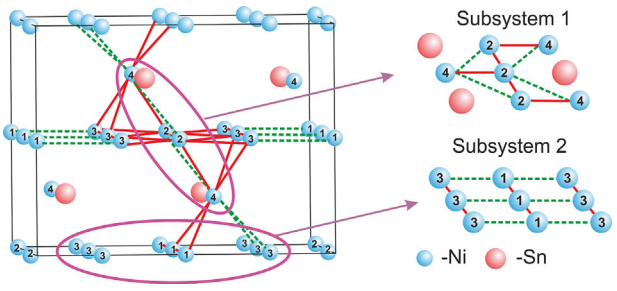

Figure 1. The positions of metallic ions in $Ni_5Sn(BO_5)_2$ crystal. Two three-lag-ladders.

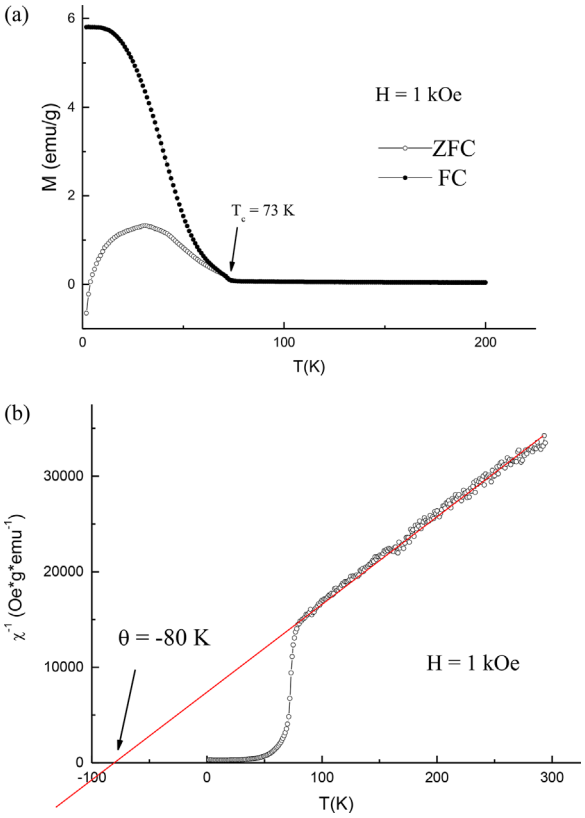

Figure 2. The temperature dependencies of magnetization in an applied field of 1 kOe (a) and the temperature dependences of inverse magnetic susceptibility (b).

The curve corresponding to the ZFC regime at low temperatures has negative values; this is an experimental artefact, and is related to the features of measurement.

The temperature dependence of the inverse *dc*-magnetic susceptibility is shown in Figure 2b; this is close to a linear law, as predicted by the Curie-Weiss law. We have $\theta_c = -80$ K. The negative value of θ_c indicates predominant antiferromagnetic

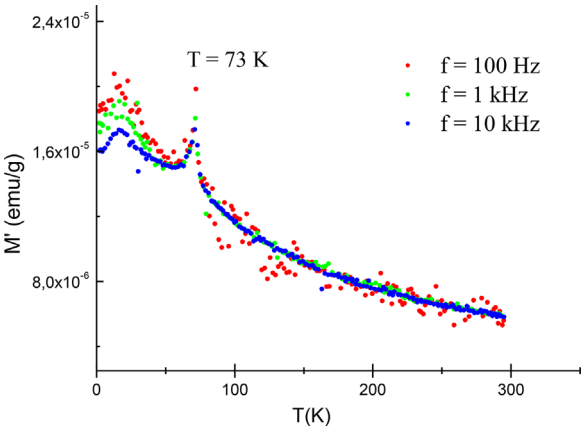

Figure 3. The real part of *ac*-magnetic susceptibility as a function of temperature.

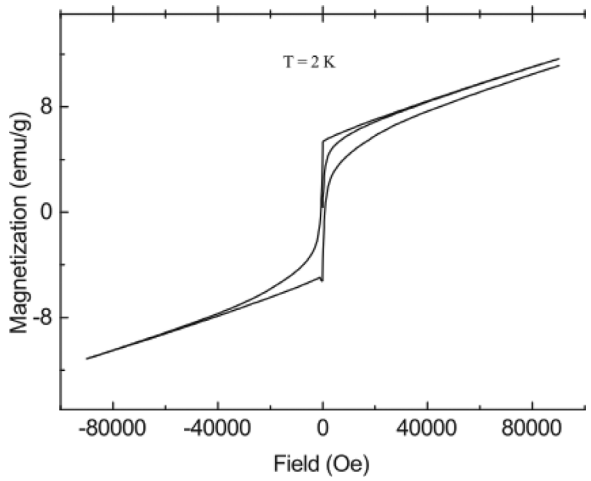

Figure 4. Magnetic field dependencies of the magnetization obtained at $T = 2$ K.

interactions. The effective magnetic moment μ_{eff} calculated for the paramagnetic phase per formula is 5.05 μ_B, and the spin component of the effective moment $\mu_s = S(S+1)$ is 4.47 μ_B. We assume that the Ni^{2+} ions are in the high-spin state, $S = 1$, and the g-factor was taken to be two. The effective magnetic moment is $\mu_J = gJ(J+1) = 14.13\,\mu_B$ per formula unit if the orbital component is taken into account. By comparing the calculated values of μ_s and μ_J with the experimental value μ_{eff}, we can see that the spin value μ_s is closer to μ_{eff} than μ_J.

The temperature dependencies of *ac*-susceptibility are illustrated in **Figure 3**. As can be seen from Figure 3, the curves have two peaks; The first peak at 73 K does not depend on the frequency of the applied field, whereas the wide peak at around 15 K does depends on this frequency. At a frequency of 100 Hz, a very wide scatter of points is observed, both at high temperatures and in the low-temperature region, although the peak at 73 K is reproduced well. In the region of the wide peak in the vicinity of 15 K, the significant scatter of points prevents us from reliably establishing whether the curve has only one feature or more. We suggest that the magnetic ordering in one subsystem appears at 73 K. When the temperature decreases, magnetic ordering begins in the other subsystem, while this subsystem stays disordered.

Figure 4 illustrates the field dependence of magnetization at 2 K. The hysteresis loop does not have a true parallelogram form, and stays open within a magnetic field of up to $H = 90$ kOe. This confirms our proposition that there is no full long-range magnetic ordering in $Ni_5Sn(BO_5)_2$.

In the future, we hope to conduct studies of single-crystal samples and to investigate the temperature dependence of the heat capacity; this will provide additional information allowing us to analyze the magnetic phase transitions in $Ni_5Sn(BO_5)_2$.

5. Theoretical Group Analysis

Theoretical group analysis was performed for the *Pnma* (62) symmetry group, which contains eight symmetry elements: a unit element, h_1; screw axes h_2, h_3, h_4; inversion h_{25}; and glide

Table 1. Eigenvectors for all irreducible representations.

		τ_1	τ_2	τ_3	τ_4	τ_5	τ_6	τ_7	τ_8
4c$_1$(2)	1	0 y 0	x 0 z	x 0 z	0 y 0	0 y 0	x 0 z	x 0 z	0 y 0
	2	0 −y 0	−x 0 z	x 0 −z	0 y 0	0 y 0	x 0 −z	−x 0 z	0 −y 0
	3	0 y 0	−x 0 −z	x 0 z	0 −y 0	0 y 0	−x 0 −z	x 0 z	0 −y 0
	4	0 −y 0	x 0 −z	x 0 −z	0 −y 0	0 y 0	−x 0 z	−x 0 z	0 y 0
4c$_2$(4)	1	0 y 0	x 0 z	x 0 z	0 y 0	0 y 0	x 0 z	x 0 z	0 y 0
	2	0 −y 0	−x 0 z	x 0 −z	0 y 0	0 y 0	x 0 −z	−x 0 z	0 −y 0
	3	0 y 0	−x 0 −z	x 0 z	0 −y 0	0 y 0	−x 0 −z	x 0 z	0 −y 0
	4	0 −y 0	x 0 −z	x 0 −z	0 −y 0	0 y 0	−x 0 z	−x 0 z	0 y 0
4b(1)	1	x y z	0 0 0	x y z	0 0 0	x y z	0 0 0	x y z	0 0 0
	2	−x −y z	0 0 0	x y −z	0 0 0	x y −z	0 0 0	−x −y z	0 0 0
	3	−x y −z	0 0 0	x −y z	0 0 0	−x y −z	0 0 0	x −y z	0 0 0
	4	x −y −z	0 0 0	x −y −z	0 0 0	−x y z	0 0 0	−x y z	0 0 0
8d(3)	1	x y z	x y z	x y z	x y z	x y z	x y z	x y z	x y z
	2	−x −y z	−x −y z	x y −z	x y −z	x y −z	x y −z	−x −y z	−x −y z
	3	−x y −z	−x y −z	x −y z	x −y z	−x y −z	−x y −z	x −y z	x −y z
	4	x −y −z	x −y −z	x −y −z	x −y −z	−x y z	−x y z	−x y z	−x y z
	5	x y z	−x −y −z	x y z	−x −y −z	x y z	−x −y −z	x y z	−x −y −z
	6	−x −y z	x y −z	x y −z	−x −y z	x y −z	−x −y z	−x −y z	x y −z
	7	−x y −z	x −y z	x −y z	−x y −z	−x y −z	x −y z	x −y z	−x y −z
	8	x −y −z	–	x −y −z	−x y z	−x y z	x −y −z	−x y z	x −y −z

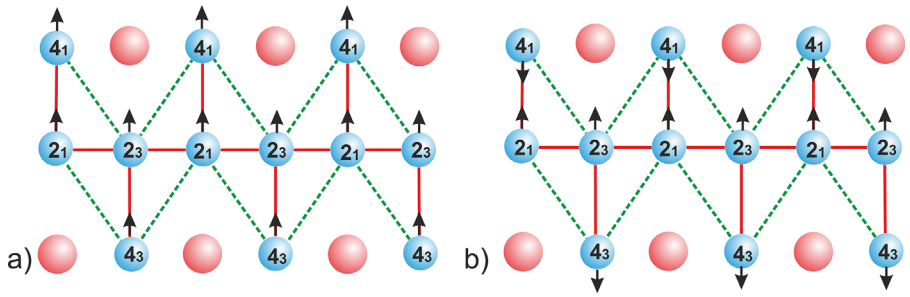

Figure 5. The possible orientations of magnetic moments in subsystem 1.

reflection planes h_{26}, h_{27}, h_{28}. The designation of symmetry operations corresponds to the work in ref. [14].

In this paper, we consider the case in which the magnetic cell coincides with the crystallographic one, and all calculations are carried out for the case of $k=0$. Magnetic representation is constructed for each element of symmetry. Then, for these magnetic representations, the decomposition of reducible representations into irreducible representations is calculated, the projection operator is constructed and its eigenvectors are calculated.

Full decomposition of reducible representation in the case of $k=0$ is as follows:

$$d^{k=0} = 7\tau_1 + 8\tau_2 + 5\tau_3 + 10\tau_4 + 7\tau_5 + 8\tau_6 + 5\tau_7 + 10\tau_8$$

Decomposition of irreducible representations for each position is as follows:

$$d^{k=0}(4b) = 3\tau_2 + 3\tau_4 + 3\tau_6 + 3\tau_8$$
$$d^{k=0}(4c_{1,2}) = 2\tau_1 + \tau_2 + \tau_3 + 2\tau_4 + 2\tau_5 + \tau_6 + \tau_7 + 2\tau_8$$
$$d^{k=0}(8d) = 3\tau_1 + 3\tau_2 + 3\tau_3 + 3\tau_4 + 3\tau_5 + 3\tau_6 + 3\tau_7 + 3\tau_8$$

The eigenvectors obtained for all irreducible representations are shown in **Table 1**. A theoretical group analysis shows that the full ordering of magnetic moments corresponds to the odd irreducible representations τ_1, τ_3, τ_5, τ_7. For the even irreducible representations, τ_2, τ_4, τ_6, τ_8, the magnetic moments of the ions in position 4b are disordered. It is notable that the magnetic moment of ions in position 4c lies along y for τ_1 and τ_5 and in the xz plane for τ_3 and τ_7, but the magnetic moments of the ions in positions 4b and 8d may have components in all (x,y,z) directions. Thus, the geometry of the structure predicts the possibility of division into two subsystems: the first formed by ions in positions 4c(4-2-4), and the second formed by ions in positions 4b and 8d (3-1-3). The orientation of the magnetic moments of the two subsystems may be noncollinear.

We now discuss the full magnetic ordering. The ferromagnetic (or ferrimagnetic) type of ordering corresponds to the τ_3(x), τ_5(y), τ_7(z) components of the eigenvectors. Other types of ordering are antiferromagnetic. There is only one type of antiferromagnetic ordering for position 4c, and therefore there are two possible types of ordering in subsystem 1 (three-leg ladder, 4-2-4). We will discuss this in the following sections. There are three different types of antiferromagnetic ordering for positions 4b and 8d.

The magnetic ordering in $Ni_5Sn(BO_5)_2$ may be complicated, and magnetic moments can become ordered at two stages: at the first stage, the partial ordering corresponds to some even irreducible representation, while at the second stage, the full ordering corresponds to some odd irreducible representation. In the following sections, we take into account only a simple model of ordering in which the magnetic moments are collinear and order by one step. In this case, there is ferromagnetic (or ferrimagnetic) ordering $(\tau_3(x), \tau_5(y), \tau_7(z))$ and one type of antiferromagnetic ordering $(\tau_7(x), \tau_1(y), \tau_3(z))$.

6. Discussion

In this section, we analyze the possible magnetic ordering in $Ni_5Sn(BO_5)_2$ using theoretical group analysis and the superexchange interactions calculated for $Ni_5Ge(BO_5)_2$ from ref. [12]. We can use the values of the superexchange interactions of $Ni_5Ge(BO_5)_2$, since both compounds are isostructural and the interatomic distances and angles of the bonds are very close. The main difference between $Ni_5Ge(BO_5)_2$ and $Ni_5Sn(BO_5)_2$ is the ordered distribution of nonmagnetic ions. In the first step, we consider "typical" ludwigite subsystems, which are two three-leg ladders, 4-2-4 and 3-1-3. The 4-2-4 subsystem contains the nonmagnetic ions. The strongest interaction in this subsystem is an antiferromagnetic 180°-superexchange interaction 4–2 (shown by the green dotted line in **Figure 5**).

Other interactions in the 4-2-4 subsystem are ferromagnetic. The theoretical group analysis gives two types of ordering for the three-leg ladder, 4-2-4. In both cases there

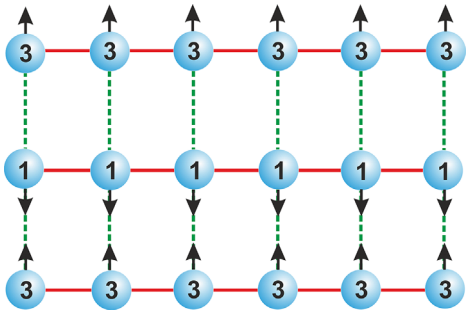

Figure 6. The possible orientations of magnetic moments in subsystem 2.

Table 2. The energies of different magnetic structures.

τ	4c1(2)[a]	4c2(4)[a]	4b(1)[a]	8d(3)[a]	Energies equations	E [K]
$\tau_3(x)$[b]	u	u	u	u	$-4J_1-4J_2-8J_3-8J_5-16J_6-4J_7-4J_8-8J_9-8J_{10}-8J_{11}$	−432.8
	d	u	u	d	$-4J_1-4J_2-8J_3-8J_5-16J_6+4J_7-4J_8-8J_9+8J_{10}-8J_{11}$	−403.2
	d	d	u	d	$-4J_1-4J_2-8J_3+8J_5-16J_6-4J_7+4J_8+8J_9-8J_{10}-8J_{11}$	−270.4
	u	d	u	d	$-4J_1-4J_2-8J_3+8J_5+16J_6+4J_7-4J_8+8J_9-8J_{10}-8J_{11}$	−6.4
	d	u	d	d	$-4J_1-4J_2-8J_3+8J_5-16J_6+4J_7-4J_8-8J_9+8J_{10}+8J_{11}$	−169.6
$\tau_1(y)$[c]	u	d	u	d	$-4J_1-4J_2-8J_3+8J_5+16J_6-4J_7-4J_8-8J_9+8J_{10}+8J_{11}$	168.8
	d	d	d	u	$-4J_1-4J_2-8J_3-8J_5-16J_6-4J_7-4J_8+8J_9-8J_{10}+8J_{11}$	−513.2
	u	u	d	u	$-4J_1-4J_2-8J_3+8J_5-16J_6+4J_7+4J_8-8J_9+8J_{10}+8J_{11}$	−226.4

[a] The orientation of the magnetic moment of the first ion in position is given in Table 2; the orientations of magnetic moments of other ions in the position should be taken from Table 1.
[b] The same ordering corresponds to $\tau_5(y)$, $\tau_7(z)$
[c] The same ordering corresponds to $\tau_3(z)$, $\tau_7(x)$

are frustrations. In the first case, antiferromagnetic interactions are frustrated, while in the second case, the 4–2 ferromagnetic interactions are frustrated. If we consider the 4-2-4 subsystem alone, the second type of ordering is preferable. For the second subsystem, 3-1-3, we can choose magnetic ordering without frustration (**Figure 6**), which agrees with the theoretical group analysis.

To find the more preferable type of magnetic ordering, we need to take into account the interaction between subsystems and the results of theoretical group analysis. We calculate the energies of the different types of magnetic ordering using the simple Ising model. We take into account the magnetic structures corresponding to the irreducible representations τ_1, τ_3, τ_5, τ_7, since in these cases, full magnetic ordering is possible. The results of this calculation are shown in **Table 2**.

The magnetic structure shown in **Figure 7** has the lowest energy. In this case, only the strongest antiferromagnetic interactions are frustrated, and although the magnetic ordering in the 4-2-4 subsystem is not preferable, other interactions compensate for the energy. Next, we look at the ludwigite structure from another point of view. The ions in positions 1, 2, and 3 form the plane. The ions in position 4 form pass of interaction between planes. In $Ni_5Sn(BO_5)_2$, the interactions between planes are weakened due to the nonmagnetic ions that occupy half positions between planes. In addition, the different interactions between ions in position 4 and ions in the plane are frustrated for all types of magnetic ordering. At the same time, we can order the magnetic moments in a plane without frustrations. It is possible that a subsystem in a plane or all moments in a plane become ordered at 73 K, and therefore when the temperature decreases the planes try to become ordered; however, there is no full magnetic order due to frustrations.

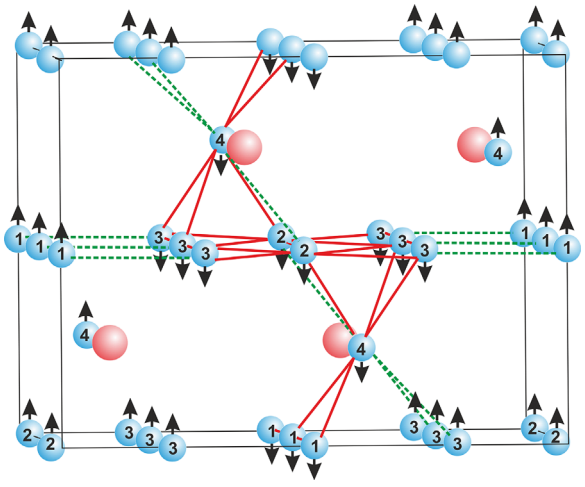

Figure 7. The possible magnetic ordering.

We calculated the temperatures of the magnetic ordering of the different subsystems,[15] taking into account the subsystem with two sublattices: 4-2-4, 3-1-3, 3-2-3. The results of the calculation are shown in **Table 3**. The 3-2-3 subsystem has the highest temperature of magnetic ordering. It is possible that the magnetic moments in the 3-2-3 subsystem order at 73 K when temperature decreases. Although ordering appears for the magnetic moments in position 1, the magnetic moments in position 4 stay disordered. This assumption contradicts the theoretical group analysis, but the magnetic moments in position 4 have fewer possibilities of ordering than those in position 1 due to frustrations.

The Ni-based ludwigites do not show a dramatic change in magnetic properties when nonmagnetic ions Sn are substituted for the Ge ions. The temperatures of magnetic ordering are similar: 87 K and 73 K for $Ni_5Ge(BO_5)_2$ and $Ni_5Sn(BO_5)_2$, respectively. We propose that the difference in magnetic properties appears due to the ordered distribution of Sn ions

Table 3. The temperature of the magnetic ordering of different subsystems.

Subsystems	T_c [K]
4-2-4	23
3-1-3	30
3-2-3	52

in $Ni_5Sn(BO_5)_2$. It is possible that superexchange interactions play the main role in magnetic ordering in Ni-based ludwigites.

7. Conclusion

Magnetic measurements of polycrystals show that there are two features at 73 K and at 15 K, corresponding to magnetic ordering. In accordance with the theoretical group analysis, the magnetic ordering may be partial or full. The magnetic structure is likely to be complicated and can be divided into two noncollinear subsystems. The antiferromagnetic structure has the lowest energy, but the ferromagnetic and ferrimagnetic structures also have low energies. Based on an analysis of theoretical and semi-empirical calculations and experimental results, we propose that there is only partial ordering in $Ni_5Sn(BO_5)_2$ due to frustrations.

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Conflicts of Interest

The authors declare no conflict of interest.

Keywords

ferrimagnets, ludwigites, magnetic phase transitions, quasi-low-dimensional structures

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