

Low temperature magnetic measurements of LiNbO₃ single crystal weakly doped with Er and codoped with Tm ions

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Abstract

Magnetic susceptibility measurements of weakly double doped lithium niobate LiNbO₃: Er (0.2 wt. %), Tm (0.3 wt. %) single crystal are reported. The dependence of the magnetic susceptibility versus temperature shows complex behavior revealing that the susceptibility may be treated as a sum of the contributions of various entities present: erbium and thulium ions, pairs and/or dimers of rare-earth ions. The results of the magnetic susceptibility measurements allow us to conclude that the distribution of rare-earth ions in weakly doped LiNbO₃: Er (0.2 wt. %), Tm (0.3 wt. %) single crystal may be strongly inhomogeneous.

Keywords: Lithium niobate; Magnetic susceptibility; Er³⁺; Tm³⁺; Rare – earth ions

1. Introduction

Erbium doped lithium niobate, LiNbO₃ (LN), has excellent perspective of applications in integrated optics and actually has been used in many technological applications [1-3]. It is a especially suitable system for solid state lasers due to non-linear properties of LN crystal [4]. Optical properties of any doped crystal are largely determined by the local symmetry of the optically active ions [5-7]. LN crystal can be tuned to fit various experiment requirements. One of the methods applied to realize this tuning is doping. Rare – earth (RE) dopant ions, e.g. Er³⁺, Nd³⁺ or Yb³⁺, are introduced to LN host as optically active impurities [8-10]. Properties of RE³⁺ centers in LN are directly related to the defects associated with the nonstoichiometric conditions (i.e. the Li/Nb concentration ratio) [11].

The constituting ions are distributed over the Li sites and the Nb ones, in most cases appreciably shifted, along the 3-fold axis, from the center of the (deformed) octahedron. There is a growing consensus that RE³⁺ ions like Er³⁺ and/or Tm³⁺ ions tend to occupy (shifted) Li-sites [2,4,12]. One may consider that within an oxygen octahedron the ionic radii of Li⁺ and Nb⁵⁺ are 90 pm and 78 pm, respectively, whereas those of Er³⁺ and Tm³⁺ are 103 pm and 102 pm, respectively. The O²⁻ radius is taken as 124 pm in 4-coordinate type: tetrahedral [13]. There are

many possibilities to attain approximate local charge compensation by locating structural vacancies in the neighborhood of a RE³⁺ ion, thus creating a large variety of optically different centers. For a discussion of the observed variety of Er sites with different surroundings one may refer to some previous papers applying electron paramagnetic resonance (EPR) technique: Nolte et al. [14] - for congruent LN: Er (0.2 mol%) and Bodziony and Kaczmarek - for LiNbO₃: Er (0.2 wt. %), Tm (0.3 wt. %) and LiNbO₃: Er (0.1 wt. %) single crystals [15-18]. One of the conclusions resulting from the above papers was that a large part of the RE-ions probably form magnetic dimers or clusters. The occurrence of dimers is supported by optical and Raman spectroscopy measurements [15-18] and site-selective spectroscopy [19]. It is difficult to choose between RE-RE dimers with RE on adjacent Li-sites, or on adjacent Li-Nb sites, although one might be inclined to prefer the latter dimer because of the local charge compensation. The temperature dependence of the EPR spectra as observed by us appears to resemble that of the observed magnetic data in the present paper.

The Er³⁺ ion (4f¹¹) is a so-called Kramers ion; with $L = 6$, $S = 1.5$, $J = 15/2$. It has a doublet ground state in the crystalline-field, split ⁴I_{15/2} lowest multiplet. The even isotopes have no nuclear magnetic moments (natural abundance 77.06 %), whereas the odd ¹⁶⁷Er isotopes have nuclear spin equal to 7/2 (natural

abundance 22.94%) [20]. The Tm^{3+} ($4f^{12}$) ion is a non-Kramers ion, with $L = 5$, $S = 1$, $J = 6$. It has a (non-magnetic) singlet as a ground state in the crystalline-field split 3H_6 multiplet. It is usually not detected in X-band EPR spectroscopy [20]. The ${}^{169}\text{Tm}$ isotope (100% natural abundance) has a nuclear spin equal to $1/2$ so the compounds containing this element possess both electronic and nuclear magnetism and exhibit Van Vleck type paramagnetism [21].

Many works have been devoted to find the preferred sites and thus the local surrounding of RE^{3+} ions in LN. The magnetic RE^{3+} dopant ions are predominantly surrounded by (distorted) octahedra of O^{2-} ions. Our single crystal is grown from a congruent melt ($[\text{Li}]/[\text{Nb}] = 0.945$) to which 0.2 wt% Er and 0.3 wt% Tm were added. Of course, this is only an input concentration. LN single crystal should have the same $[\text{Li}]/[\text{Nb}]$ ratio with about 4.6% vacant Nb^{5+} (or Li^+) sites and about 0.6 mol% RE ions. Assuming a random distribution we could expect that majority of the RE^{3+} -ions (moments) is not 'exchange' coupled to any other RE^{3+} moment. The same is true for RE^{3+} - RE^{3+} pairs (dimers) or clusters that may be formed preferentially during the preparation: also these clusters should, at first instance, be regarded as single entities, not exchange coupled to other 'entities'. Other, long-range coupling mechanisms (such as dipole-dipole interaction, or even more exotic ones like magneto-elastic or magneto-electric (-elastic) coupling) are not supposed to give rise to ordering in temperatures above, say, 1 K. For a system of magnetically coupled, identical 'free' moments the Curie-Weiss (CW) law for the susceptibility holds: $\chi = C/(T-\theta)$. Here the CW temperature, θ , is a measure for the coupling (say, the Weiss molecular-field parameter). When the moments are placed in a crystalline field with an uniaxial component [4], a shift in θ may occur. The value of the θ parameter as well as the shift in θ is of the order of 1 K (see for instance Gruber et al., Er^{3+} in Er_2O_3 and Er^{3+} in Y_2O_3 , [22]). From the crystalline-field calculations and magnetic susceptibility measurements for systems with Er^{3+} ions in comparable (oxygen octahedron) surroundings, one can conclude that, in general, the Weiss molecular-field coupling is the minor importance. Magnetic ordering is not found for temperatures above about 1 K. Reasonable CW-like behavior is observed in a large temperature interval. Also for the non-Kramers Tm^{3+} ion the single-ion approach does work satisfactorily in analogous surroundings (see Awaka et al. [23]). In the Tm garnet $\text{Tm}_3\text{Al}_5\text{O}_{12}$, a constant (Van-Vleck) susceptibility is observed up to about

20 K. At higher temperatures, the susceptibility decreases eventually showing more or less CW behavior [23]. In this case, any derived CW-temperature must be interpreted with great caution [24].

We may conclude that, at first instance, the single-entity approach is suitable for RE^{3+} ions and clusters in LN as well. Of course, as mentioned above, the RE^{3+} ions can have many different surroundings, each of them leading in principle to a different CW term, with a different CW-temperature. When, however, the $|\theta|$ -values are below about 2 K, the resulting $1/\chi$ vs. T curve is quite close to a straight line in our measuring interval 4 K – 40 K [15-18]. As argued above, in the first approximation, also the clusters can be treated as independent entities. One way to obtain any estimation is the Bleaney-Bowers approach [21,25], widely used for the analysis of magnetization data observed for 'dimers'.

To our knowledge there are not so many publications giving susceptibility versus temperature data for (doped) LN. Díaz-Caro et al. [26] did measure the magnetic susceptibility of LN doped with Cr^{3+} . Bodziony et al. [16,17] measured the susceptibility in the temperature range 4 K – 40 K for LN: Yb (1wt%) and LN: Yb (0.8wt%) Pr(0.1wt%). In all these cases approximate CW behaviour was observed, with estimated CW temperatures of the order of 1 K again.

The paper is organized as follows: in the Experimental section, the material and methods applied are presented. The magnetization measurements for weakly doped LN: Er (0.2 wt. %) and Tm (0.3 wt. %) crystals are presented in the third part. Finally we discuss the obtained results.

2. Experimental

LiNbO_3 single crystal doubly doped with Er^{3+} (0.2 wt. %) and Tm^{3+} (0.3 wt. %) was grown, along the c – axis, from the congruent melt by the Czochralski method in the Institute of Electronic Materials Technology, Warsaw, Poland [27]. After mixing of adequate amounts of reagents the mixture was calcined at 1373 K for 6 h. In order to obtain a polar single-domain crystal an electric field was applied for 30 min. at 20 °C above the Curie temperature (ca 1140 °C). Afterwards the crystal was cooled down to room temperature for 24 hours. The electric field was switched off when the temperature decreased down to 1050 °C [27]. The ingredients: Er_2O_3 and Tm_2O_3 were added to the congruent melt with a Li/Nb ratio equal to 0.945, prior to synthesis at elevated temperatures.

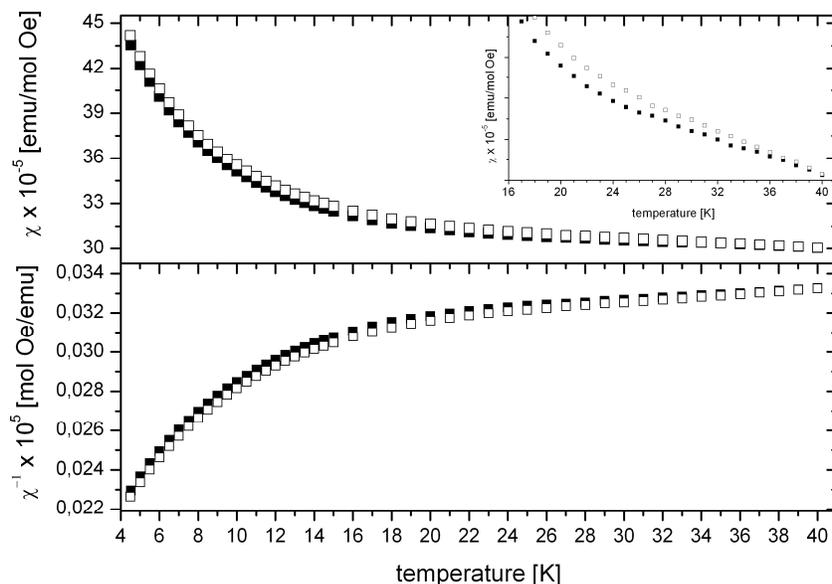


Figure 1. The upper figure shows the temperature dependence of the magnetic susceptibility, χ , for LN: Er (0.2 wt. %), Tm (0.3 wt. %) single crystal in ZFC and FC regimes (solid squares and empty squares, respectively); the lower figure presents the temperature dependence of the inverse magnetic susceptibility $1/\chi$ (lower panel). The inset shows the MS measurements in the high-temperature region.

The magnetization of the single crystal was measured in the temperature range from 4 K to 40 K using a Quantum Design magnetometer. The sample was cooled, at first, without magnetic field (zero field cooled, ZFC) and the measurements were carried out with temperature increasing from 4 K up to 40 K in an applied magnetic field $H = 200$ Oe. The direction of the magnetic field was chosen to be parallel to the optical z -axis (crystallographic c -axis) of the LN unit cell. Subsequently, the sample was cooled down to 4.5 K in a magnetic field of 200 Oe (field cooled, FC) and the measurements were carried out with temperature increasing up to 40 K in the same magnetic field. The measurements were performed at Institute of Nanotechnology, Karlsruhe, Germany.

3. Magnetic susceptibility measurements

Figure 1 shows the magnetic susceptibility ($\chi = M/H$) versus temperature (upper figure) and the temperature dependence of the inverse magnetic susceptibility ($1/\chi$, lower figure) for the LN: Er (0.2 wt. %), Tm (0.3 wt. %) single crystal in ZFC and FC regimes (solid squares and empty squares, respectively). The inset in Figure 1 shows the change in the temperature dependence of the magnetic susceptibility in the temperature range above approximately 16 K. As one can see, the ZFC

and FC points lie enough far away from each other to be clearly distinguished.

The rather complex behaviour can be described by recognizing two temperature regions: the low-temperature region, below ≈ 16 K, and the high-temperature region, above ≈ 16 K. In the first region, temperatures up to ≈ 16 K, the susceptibility data can be described by:

$$\chi_1 = \chi_1^0 + \frac{C_1}{T - \theta_1} \quad (1)$$

where χ_1^0 is a constant term (temperature independent paramagnetism – TIP or Van Vleck paramagnetism). Figure 2 presents the temperature dependence of the product of the temperature and the magnetic susceptibility (less TIP term) versus temperature for LN: Er (0.2 wt. %), Tm (0.3 wt. %) single crystal in ZFC regime. The analogous dependence for FC regime is very similar and only a few shifted.

One can observe two maxima of the $(\chi - \chi_0)T$ dependence vs. temperature. First maximum occurs at low temperature: $T = 7.0 \pm 1.0$ K, and the second one at higher temperature, $T = 38.0 \pm 1.0$ K. According to our previous EPR measurements of LN: Er and LN: Er, Tm single crystals [16,17], we suggest that the two maxima visible at low and high temperatures (see Figure 2) could be

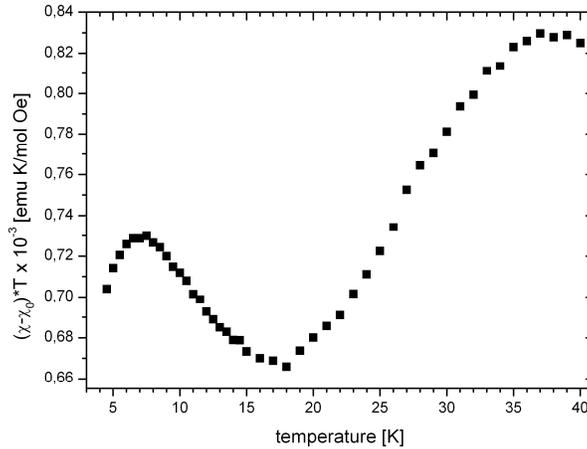


Figure 2. The temperature dependence of the $(\chi - \chi_0)T$ versus temperature for LN: Er (0.2 wt. %), Tm (0.3 wt. %) single crystal in ZFC regime.

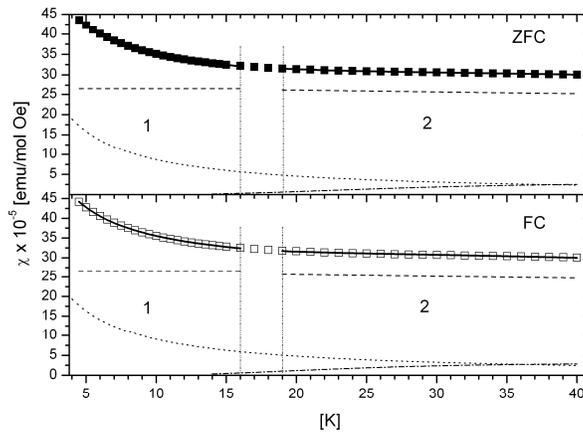


Figure 3. The temperature dependence of the magnetic susceptibility measured for LiNbO₃: Er (0.2 wt. %), Tm (0.3 wt. %) single crystal in ZFC (upper panel, solid squares) and FC (lower panel, empty squares) regimes, respectively, with fitted curves (according to Eqs 1 and 2) in the low and the high temperature regions. Dashed: alleged Tm contribution, dotted: Er³⁺ Curie-Weiss contribution; dash-dotted: Bowers-Bleaney Er³⁺-Er³⁺ dimer contribution; solid: total.

attributed to the Er³⁺ and Tm³⁺ ions, respectively. Similarly, the CW term in Eq. 1 can be attributed to the Er³⁺ moments. In line with the discussion in the introduction, we may assume that this term holds also for the higher-temperature range. The constant term is thought to be mainly due to the Van Vleck paramagnetism of the Tm³⁺ ions. In the higher temperature region one may expect a decrease of this Tm³⁺ contribution that we tried to approximate roughly by a CW term, which value, at $T \approx 16$ K, is equal to the constant term value of Eq. 1. We tried also use a fitting procedure in which the alleged Tm³⁺ Van Vleck contribution is kept constant in the high-temperature region. In any case an extra term

rising with temperature increase is necessary. As stated in the introduction, for this purpose we added a Bleaney-Bowers term ($S = 1/2$) [25], and adopted, in the higher temperature region, the following equation:

$$\chi_2 = \frac{C_1}{T - \theta_1} + \frac{C_3}{T - \theta_3} + \frac{C_{BB}}{T} \left(1 + \frac{1}{3} \exp\left(\frac{-2J_w}{kT}\right) \right)^{-1} \quad (2)$$

Notice that a possible constant contribution was neglected in order to reduce the number of free parameters. Figure 3 presents the temperature dependence of the same magnetic susceptibility data obtained in ZFC (upper panel,

Table 1. Fitting parameters for the low-temperature region (see eq. 1).

	χ_1^0 [10^{-4} emu/mol Oe]	θ_l [K]	C_1 [10^{-4} emu K/mol Oe]
ZFC	2.65 ± 0.01	-1.1 ± 0.1	9.6 ± 0.2
FC	2.65 ± 0.01	-1.2 ± 0.1	10.2 ± 0.2

solid squares) and FC regimes (lower panel, empty squares) with fitted curves for the low-temperature (below 16 K, marked by 1) and the high-temperature (above 16 K, marked by 2) regions, according to Eqs 1 and 2, respectively. The Bleaney – Bowers term decreases rapidly to zero at about 14 K, hence the function is shown in the whole temperature range up to a temperature of 40 K (see Figure 3). Vertical dash-dot lines designate the upper bound of the low-temperature region and the lower bound of the high-temperature one, 16 K and 20 K, respectively. The obtained fitting parameters are collected in Tables 1 and 2.

As one can see, the fitting of the CW function for the alleged Tm^{3+} contribution leads to an unphysical result, because of the large value of the obtained CW temperature (see Table 2). We may remark that in a system of coupled moments of different kinds a more intricate relationship holds than the CW law used in eq. 1. Thus, the susceptibility can be decomposed in a sum of CW-like terms in each of which the CW-temperature is some expression of all the coupling and ion parameters. Formally the derivation is analogous to that for a ferrimagnet, presented in many textbooks.

As said above, we also tried to perform a fitting procedure to the MS data, using the same, constant value for Tm^{3+} contribution in both temperature regions. As expected, the obtained fitting constants for the Bowers-Bleaney term do not very much differ: $C_{BB} \approx 40 \times 10^{-4}$ emu K/mol Oe and is somewhat lower of course, and $J_w \approx 29$ cm⁻¹ remains the same. The fitting function for the alleged Tm^{3+} contribution could be treated as an artificial

approximation only, serving to estimate the order of magnitude of the Bleaney-Bowers ‘correction’.

There is possible one more explanation of the observed magnetization measurements, taking into account excited states of Er^{3+} ions. We will analyze this possibility in the near future.

4. Discussion

We have found only few examples of applying of the Curie – Weiss law to paramagnetic ions in a diamagnetic matrix. J. Kliava et al. have applied the Curie – Weiss term (with additional Curie term) to description of Gd^{3+} ions in multicomponent oxide glasses, where gadolinium was added in quantities from 0.1 to 10 mass % [28]. They deduced the presence of the Gd^{3+} clusters in multicomponent oxide glasses [28]. D.M. Gill, L. McCaugham and J.C Wright investigated spectroscopic sites in erbium doped lithium niobate [29]. They concluded that increasing the dopant concentration tends to increase the Li deficiency in the LN crystal and reduces the cluster site concentration. Taking 1.0 mol % of $LiNbO_3:Er$ crystal close to stoichiometry, i.e. reducing Li_2O deficiency, they have found the cluster site concentration to be reduced by about ~ 30%. However the absorption intensity of the light in the LN crystal was raised [29]. Consequently, in the case of low erbium concentration (for example 0.2 or 0.3 %) the cluster site concentration should be significant. The paper by J. Diaz-Caro et al. [26] was particularly discussed previously, in the Introduction.

Table 2. The fitting parameters according to Eq. 2 in the high temperature range (16 K < T < 40 K).

	C_3 [10^{-4} emu K/mol Oe]	θ_3 [K]	C_{BB} [10^{-4} emu K/mol Oe]	J_w [cm ⁻¹]
ZFC	0.147 ± 0.024	-544 ± 93	39.8 ± 3.4	-30.2 ± 0.5
FC	0.143 ± 0.107	-541 ± 120	36.0 ± 11.9	-26.1 ± 3.2

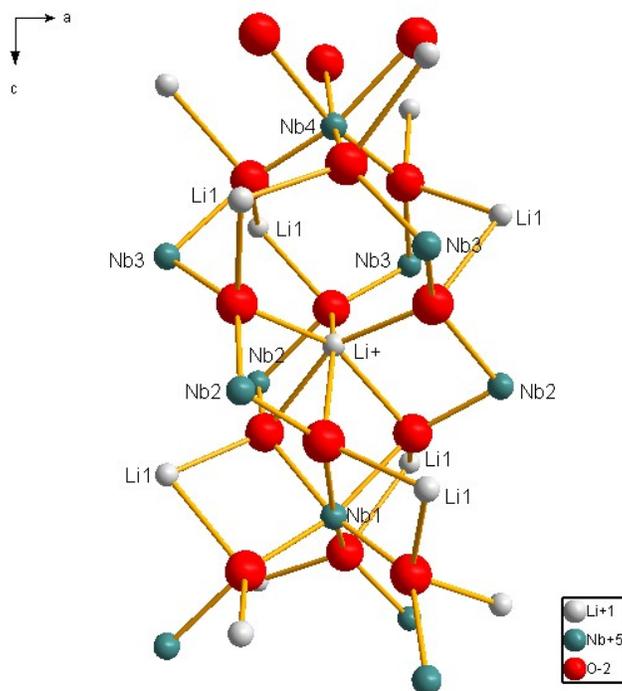


Figure 4. A schematic view of the Li^+ ion environment in the LiNbO_3 single crystal.

Returning to our measurements, we firstly discuss the fitting results in the low-temperature region. The splitting in a constant term and a CW contribution (see Eq. 1) appears to be physically acceptable. The constant term is interpreted as a sum of contributions like the diamagnetic susceptibility and the low-temperature (single-ion) Van Vleck contributions. There may also be a “constant” contribution of the (e.g. antiferromagnetic Er-Er dimers) clusters, but we ignore such a contribution by adopting the Bleaney-Bowers description for the ‘dimers’. We used the magnetization data obtained by Diaz-Caro et al. [26]. From the experimental data we estimated the diamagnetic susceptibility χ_{dia}^0 for LN:Cr, by “imposing” a generalized Curie-Weiss behaviour $(\chi - \chi_{\text{dia}}^0)^{-1} = (T - \theta) / C$ (see Figure 2). From the data for zero Mg concentration, we could fit this relation reasonably with $\chi_{\text{dia}}^0 \approx -0.6 \times 10^{-4}$ emu/mol Oe, and, $\theta = 3$ K. This would raise the ‘Van Vleck’ contribution to $(\chi_1^0 - \chi_{\text{dia}}^0) = (2.65 + 0.6) \times 10^{-4} = 3.25 \times 10^{-4}$ emu/mol Oe.

The fitting results in the high-temperature region are more complex. The magnetic susceptibility reveals as sum of three terms: two CW terms, and Bleaney-Bowers term

(see Eq. 2). One CW term is the same as in the low-temperature region and originates from the Er^{3+} ions. The second CW term is the one, which was constant (diamagnetic) term in the low-temperature region. This term is attributed to the Tm^{3+} ions. It seems that the Bleaney-Bowers term describes the ‘dimers’ of magnetic ions in LN host, both Er^{3+} and Tm^{3+} ions.

To understand correctness of the above assumptions we need a closer look at the lithium ion environment (the environment of niobium ion is similar). Figure 4 shows the schematic view of lithium ion (Li^+) environment in the LN single crystal, assuming that the center of the structure is at the Li^+ ion. The dopant ions (Er^{3+} or Tm^{3+}) in the LN host can be substituted for Li^+ or Nb^{5+} sites. The sequences of Li^+ , Nb^{5+} and vacancy octahedrons along the C_3 axis in LN are connected through oxygen’s bridge (O^{2-}). The magnetic ions can interact generally in two ways: through space or through bonds [30]. Assuming that Li^+ site is substituted by RE^{3+} (Er^{3+} or Tm^{3+}) ions, there are about 14 sites being possible to substitute for other RE^{3+} ions in the closer neighborhood. Two of RE^{3+} ions may interact with each other through bonds or through space because the ions are close to each other [30]. The presence of different possible exchange interactions between the RE moments makes the situation even more complex.

MS measurements of LN: Er (0.2 wt. %), Tm (0.3 wt. %) sample reveals the presence a few magnetic systems in LN: Er, Tm single crystal. Our conclusions are summarized in the following points:

MS measurements seem to be dominated by signal originating from Tm^{3+} ion in all temperature range. We suggest, that diamagnetic terms in the low temperature region (Van Vleck type paramagnetism) and the Curie – Weiss term in high temperature region can be attributed to Tm^{3+} (see Figure 3, dashed lines). The behavior of the magnetic susceptibility term originating from Tm^{3+} ions (Figure 3, dashed lines) may be regarded as low-temperature approximation (up to 40 K) for the highly complex functions from Awaka et al. [23]. We can determine the quantitative proportions taking the data from the paper [23]. Magnetic susceptibility of $\text{Tm}_3\text{Al}_5\text{O}_{12}$ single crystal is equal to (low-temperature limit): $\chi = 0.2$ M/H/emu (per mol Tm^{3+} ion). Theoretical values of MS are in the range from $\chi = 0.08$ to $\chi = 0.23$ M/H/emu (mol Tm^{3+})⁻¹ [23]. The ratio of χ/a_1 is equal to 755 (for experimental value) and locates in a range between 302 and 870 (for theoretical values). The MS signal should be proportional to the number of Tm^{3+} ions in LN single crystal. Assuming that all of Tm ions entered the LN lattice, the ratio of Tm ions in both crystals is equal to 333. This value is within theoretical limit. It is a reasonable result, which may suggest that most of Tm ions entered LN: Er (0.2 wt. %), Tm (0.3 wt. %) single crystal during the manufacturing process. So, the results are qualitatively and quantitatively consistent with those given by Awaka et al. [23].

Erbium ions give probably the second largest contribution to the MS signal in LN: Er (0.2 wt. %), Tm (0.3 wt. %) sample. It is represented by Curie Weiss term in all temperature region (see Figure 3, dotted lines). A negative value of the Curie temperature $\theta_f = -1.2$ [K], suggests the presence of antiferromagnetically interacting Er^{3+} ions. Our previous EPR measurements confirm these predictions [17,18]. Figure 3 shows that MS signal from erbium ions is weaker than the signal from the thulium ions. This is consistent with the input concentrations of these elements.

The presence of the Bleaney - Bowers (BB) term is rather a surprise. The best fit can be obtained without a relevant BB-contribution, using for example an important contribution of an excited Er level. This is a very complex problem from a theoretical point of view. We decided to use a BB term as a first approximation to solve this problem. The Bleaney – Bowers equation usually is used to describe the dimers (cluster) in exchange models,

for example Cu^{II} dimers in many compounds [31,32]. Figure 3 reveals that Bleaney – Bowers term decreases rapidly to zero in low temperature region (at ~ 14 K) but becomes even bigger than the Curie – Weiss term in a temperature ~ 40 K (dash-dot line, Figure 3) and next decreases above 40 K. The presence of Bleaney – Bowers term gives much less error of the fitting procedure than the same procedure without this term. The existence of Bleaney – Bowers term suggests, that there are dimers of RE^{3+} ions (Er^{3+} and/or Tm^{3+}) in LN: Er (0.2 wt. %), Tm (0.3 wt. %) single crystal, proving a weak antiferromagnetic exchange interaction ($J_w = -26$ [cm⁻¹] or -37 K). The occurrence of dimers (clusters) of RE^{3+} ions in LN single crystals is confirmed by optical and Raman spectroscopy measurements, site-selective spectroscopy and our previous EPR measurements.

We could not record magnetic signals originating from isolated magnetic ions. Taking into account a very low concentration of dopant ions we suggest that the distribution of the dopant ions in weakly doped LN: Er (0.2 wt. %), Tm (0.3 wt. %) crystal may be strongly inhomogeneous. The reasons may be the crystal structure (the presence of structural vacancies) and the charge compensation mechanism. It seems that erbium and thulium ions easily form clusters (dimers) in LN lattice despite a weakly dopant concentrations. The conclusion is consistent with the results presented by D. M. Gill et al. [29] and our previous EPR results [17,18].

5. Conclusions

In this paper we present only a first approximation to solve a problem of magnetic susceptibility measurements of LN: Er (0.2 wt. %), Tm (0.3 wt. %) single crystal. The magnetic measurements of weakly (separately) doped LiNbO_3 : Er and LiNbO_3 : Tm single crystals are necessary to obtain more accurate explanation of the behavior of magnetic moments in weakly doped LN: Er, Tm single crystals.

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