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Antiferromagnetic resonance study of the magnetic structure of $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$

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The effect of replacing Nd^{3+} ions with Dy^{3+} ions on the resonance properties of $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ is studied. The antiferromagnetic resonance (AFMR) method is used to detect an easy axis-easy plane magnetic spin-reorientation phase transition owing to competing exchange interactions of Nd–Fe and Dy–Fe, and the frequency-field dependence of the AFMR spectrum before and after the phase transition is studied. At 4.2 K, the detected resonance spectra in the $\mathbf{H}\|\mathbf{c}$ direction are AFMR modes of iron, and their frequency-field dependences correspond to easy-axis ($H < 15$ kOe) and easy-plane ($H > 16$ kOe) magnetic structures with magnon excitation energy gaps of 77.2 and 100.3 GHz, respectively. The effective magnetic anisotropies responsible for these gaps are 0.7 and 1.2 kOe, respectively. Substitution by Dy^{3+} ions in crystalline $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ in the paramagnetic region produces an additional static internal field owing to polarization, which leads to a shift in the resonance field of the EPR absorption line and a change in the *g*-factor of the Fe^{3+} ions. © *2014 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4890992]

The magnetic, electrical, and magnetoelectrical properties of the rare earth ferroborates $RFe_3(BO_3)_4$, which belong to the $R32$ trigonal space group, are currently under intense study. Two interacting ordered subsystems (magnetic and electrical) exist simultaneously in these systems.[1–4] The spins of the Fe^{3+} ions are ordered antiferromagnetically at temperatures of 30–40 K, and their mutual orientation is influenced by the paramagnetic subsystem of the rare earth ions R. Depending on the anisotropy of the rare earth ions, the R–Fe exchange interaction stabilizes either the easy-plane magnetic state in the *ab* plane of the crystal or the uniaxial states along the *c* axis.[5–8] By replacing the rare earth ions by other isovalent ions in the non-centrally symmetric rare earth ferroborates $RFe_3(BO_3)_4$ it is possible to make significant changes in their magnetic and electrical properties, as well as in the relationship between these properties. The competition between the magnetic anisotropy of substituted rare earth ions can lead to the appearance of spontaneous (with changes in temperature) and induced (by a magnetic field) spin-reorientation magnetic phase transitions.[9–14]

A large amount of experimental data has been gathered in the course of research on the substituted ferroborates, in particular $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$.

Temperature and field dependences of the magnetic susceptibility, magnetization, and specific heat of the mixed compound $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ are reported in Refs. 9–11. Spin-reorienation transitions were observed with respect to magnetic field and temperature. The experimental results differ little among themselves, but they do confirm the complexity of the behavior of the magnetic subsystem of this crystal in external magnetic fields or at low temperatures. The low-temperature magnetic phase transitions have been studied[12] in more detail using an ultrasonic technique and an *H–T* phase diagram was constructed. During the

resonance measurements it was found that our results are in good agreement with the data of Ref. 12.

Raman and Brillouin scattering of light, antiferromagnetic resonances (AFMR), and other techniques have been used very effectively for studying the spin oscillations in multiferroics.

The antiferromagnetic resonance technique has been used[13,14] to study the systems $YFe_3(BO_3)_4$–$GdFe_3(BO_3)_4$–$Y_{0.5}Gd_{0.5}Fe_3(BO_3)_4$ and $ErFe_3(BO_3)_4$–$Er_{0.75}Tb_{0.25}Fe_3(BO_3)_4$ in which AFMR modes of the Fe^{3+} ion subsystem are observed. The frequencies were found to depend strongly on the character of the magnetic anisotropy of the rare earth ions and their exchange interactions with the Fe-subsystem. The major parameters of the magnetic interactions were determined.

This paper is a study of the effect on the resonance properties of $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ of replacing Nd^{3+} ions with Dy^{3+} ions using antiferromagnetic resonance techniques over wide ranges of frequencies, temperatures, and external magnetic fields. The magnetic and resonance properties of this kind of ferroborate are evidently determined by both the antiferromagnetic Fe-subsystem and the two types of paramagnetic rare-earth ions (Nd^{3+} and Dy^{3+}), which exist in the external magnetic field and in the exchange field of the Fe^{3+} ions. There are no published data from resonance studies of this compound.

In an earlier paper[15] we studied the resonance properties of crystalline $NdFe_3(BO_3)_4$ in the ordered state. A detailed study of the AFMR frequency-field dependences showed that the magnetic moments of the Nd and Fe lie in the *ab* basis plane, while the observed resonance modes correspond to oscillations of an antiferromagnetic structure with an "easy plane" anisotropy and a weak anisotropy in the basis plane of the crystal, and made it possible to determine the

effective magnetic parameters (the magnetic anisotropy and the sizes of the energy gaps) for the magnetic structure of $NdFe_3(BO_3)_4$.

In crystalline $DyFe_3(BO_3)_4$ the magnetic moments of the Fe^{3+} and Dy^{3+} ions are oriented along the trigonal *c* axis—an easy-axis antiferromagnet with trigonal symmetry. We shall clarify how the magnetic structure and effective magnetic parameters change as Nd^{3+} is replaced by Dy^{3+} in $NdFe_3(BO_3)_4$.

The resonance properties of $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ were studied with a standard direct amplification radio spectrometer in stationary magnetic fields of up to 75 kOe. Resonant absorption was excited by perpendicularly polarized microwave radiation. Samples with sizes of roughly $2 \times 3 \times 2$ mm grown by the technique described in Ref. 16 were used in the measurements. The samples were oriented using an x-ray method.

Figure 1 is an H–T phase diagram for a field orientation $\mathbf{H} \| \mathbf{c}$ (*z* axis).[12] Here curves 2 and 3 are treated as the curves for the orientational magnetic phase transitions. It has been claimed[9–14] that the compound $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ has an easy-plane magnetic structure below $T_N = 32$ K (line 1) down to the phase transition curve 2. There is an intermediate phase with fixed magnetic structure within the temperature interval from 26 to 16 K. At temperatures below the transition curve 3, this system is in a low-temperature easy-axis magnetic phase.

The first resonance measurements were made for low frequencies at 4.2 K. Figure 2 shows the field traces of the spectrum for several frequencies. It is clear that at 40.07 GHz there is no resonance spectrum and the observed two-step anomaly in the form of jumps at a similar magnetic field undergoes hysteresis. The magnetic fields at which the jumps occur ($H_1 = 14.920$ Oe, $H_2 = 15{,}850$ kOe) correspond to a spin-reorientation magnetic phase transition through an intermediate phase. The orientational transition is a first order transition. The magnitude of the intermediate phase with respect to magnetic field in our experiment is 930 Oe.

It is assumed that spin-reorientation transitions in which an easy-axis magnetic structure becomes an easy-plane structure are being observed. At high frequencies the jumps look somewhat different, but appear at the same magnetic fields, which confirms the scenario of phase transitions. As the temperature is lowered to 1.7 K the magnitude of the
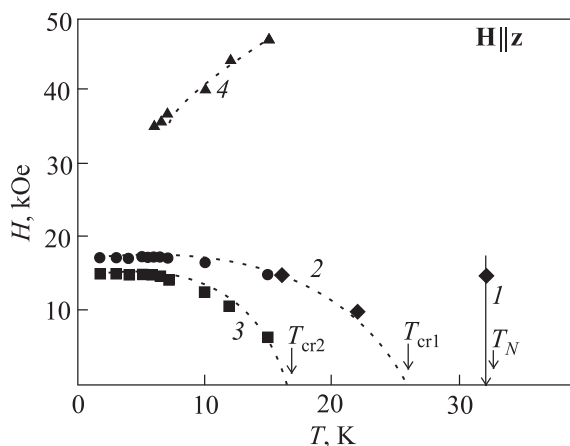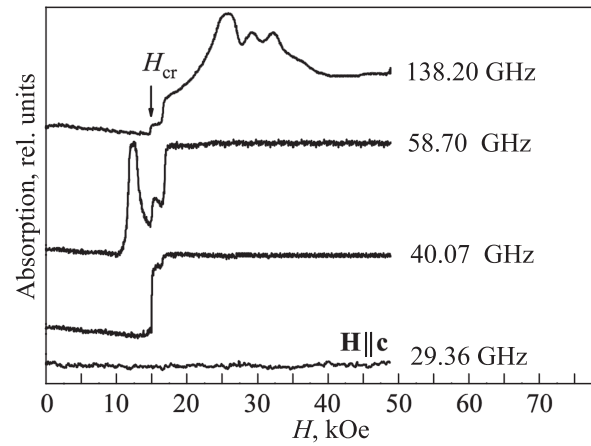

FIG. 2. Field traces of the spectrum of crystalline $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ for different frequencies ($\mathbf{H} \| \mathbf{c}$, $T = 4.2$ K).

fields for the transitions remain unchanged to within the error.

Over the entire range of frequencies that were measured, no spin-reorientation phase transitions were observed in magnetic fields of up to 50 kOe with an $\mathbf{H} \perp \mathbf{c}$ orientation (See Fig. 2).

Based on these measurements we can state that the magnetic structure in crystalline $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ undergoes a readjustment in an applied magnetic field, i.e., for $\mathbf{H} \| \mathbf{c}$ a spin-reorientation phase transition takes place through an intermediate phase at low (4.2 K) and ultralow (1.7 K) temperatures. It was assumed in Refs. 9–14 that an easy-axis magnetic structure exists in the lowest temperature region of the phase diagram.

To confirm these conclusions we have attempted to observe the AFMR spectrum experimentally and to determine its frequency-field dependence along the **c** axis. Figure 3 shows the shape of the AFMR spectrum and its transformation with changing frequency and magnetic field. There are clearly two branches of the oscillations in fields up to 15 kOe which converge in zero magnetic field for a magnon excitation frequency of 77.2 GHz. In nonzero magnetic field the frequencies of the branches vary in direct proportion to the strength of the external magnetic field.
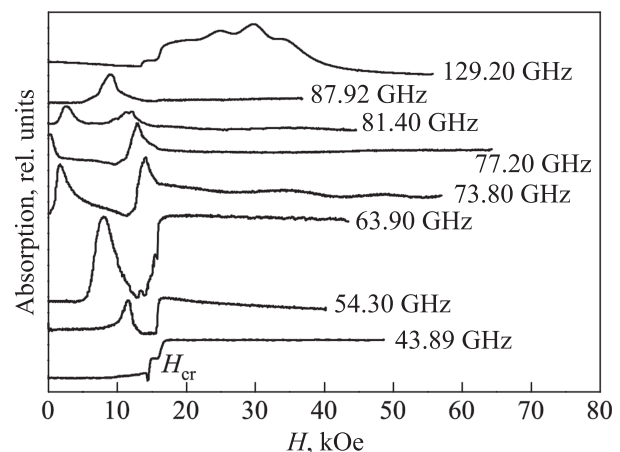

FIG. 1. H–T phase diagram of $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ (Ref. 12) (see text).

FIG. 3. The AFMR spectrum of the compound $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ and its transformation as the frequency and magnetic field are varied for the easy-axis magnetic structure ($\mathbf{H} \| \mathbf{c}$, $T = 4.2$ K).

The shape of the AFMR spectrum for fields above 16 kOe and **H**||**c** is shown in Fig. 4. Very broad lines are observed with structures that are complicated to interpret. We note that our discussion applies to two-sublattice antiferromagnets, while the actual $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ has multiple sublattices and its unit cell contains different species of magnetic ions. Factors such as a high excitation frequency, a large number of sublattices, and the possible formation of clusters can lead to the appearance of resonance lines with complicated structures.

The frequency-field curves for **H**||**c** are linear and are satisfactorily described by the equation for uniaxial antiferromagnets, $\vartheta_{1,2}/\gamma = \sqrt{2H_eH_a} \pm H$ (Fig. 5). This means that the antiferromagnetism vector **l** || **c**. Given the magnitude of the exchange interaction (540 kOe) and our experimentally determination of the energy gap (77.2 GHz), we obtain an effective anisotropy of $H_A = 0.7$ kOe.

For a magnetic field oriented along the **a**(**H** || **a**⊥**l**) axis, at an experimental temperature of 4.2 K there is an AFMR branch that depends quadratically on the external magnetic field. In zero field the AFMR excitation energy is 77.2 GHz. The frequency-field dependences for this mode are shown in Fig. 5. No other resonance bands are observed, and the AFMR spectrum for fields below 15 kOe corresponds to a uniaxial magnetic structure with an easy-axis anisotropy parallel to the principal axis of the crystal (the **c** axis).

Observation of the AFMR spectrum for a uniaxial magnetic structure is limited by phase transition curve 3 in the phase diagram (Fig. 1).

In magnetic fields higher than 16 kOe and at 4.2 K, the two branches typical of a collinear uniaxial structure vanish and are replaced by a branch with an initial splitting at zero field of 100.3 GHz (Fig. 5). The magnitude of the anisotropy that produces this gap is 1.2 kOe. The excitation frequency of the AFMR branch increases quadratically with increasing magnetic field.

For a magnetic fields (**H**⊥**c**) lying in the basis plane, there is no spin-reorientation phase transition in the available external fields for a temperature of 4.2 K (Fig. 2); thus, no AFMR spectrum is observed.

Therefore, as can be seen from Fig. 5, for magnetic fields of 15 > H > 16 kOe with orientation **H**||**c** at T = 4.2 K,

FIG. 5. Frequency-field diagram of the AFMR spectra of crystalline $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ for easy-axis and easy-plane magnetic structures (**H** || **c** and **H** ⊥ **c**, T = 4.2 K).

highly different frequency-field AFMR dependences can be observed in the Dy^{3+} substituted ferroborate $NdFe_3(BO_3)_4$; these are evidence of magnetic-field induced spin-reorientation phase transitions from an easy-axis to an easy-plane magnetic state.

According to the phase diagram, the easy-axis magnetic structure is retained from 1.7 to 16 K. As the temperature is increased to 16 K, a decrease in the field of the spin-flop transition is observed; this is not typical of uniaxial antiferromagnets. Here it becomes more difficult to observe AFMR since the phase-transition field decreases with rising temperature, while there is a decrease in the range within which AFMR modes are excited.

We note that the critical field for the spin-flop transition can decrease with rising temperature owing to a reduction in the total effective anisotropy constant of a compound, which may indicate an instability in the collinear phase with rising temperature, while the parallel susceptibility increases.

The magnetic structure of $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ is indeterminate for temperatures in the range 16–26 K.[10–14] It can be assumed that this is an intermediate phase between two magnetic phase transitions, although the temperature interval for the intermediate phase is slightly wider than usual. In the resonance measurements (T = 4.2 K) the magnetic field interval between two jumps was only 930 Oe, which corresponds fully to the intermediate phase for a first order phase transition. Unfortunately, the antiferromagnetic resonance does not give a unique answer to this question at temperatures of 16–26 K. Within this temperature interval, very broad lines are observed which cannot be determined and used to construct the frequency-field dependences.

We believe that the data of Ref. 17 and our resonance measurements show that the Fe^{3+} ion subsystem is in a collinear phase for T = 4.2 K and H = 0. In the resonance measurements, a two-step anomaly in the form of sharp jumps is observed as the external magnetic field is increased; the magnetic field for these jumps coincides with that of the jumps in the magnetization. Analysis shows that the jumps are caused by transitions from the collinear phase to a flop-phase of the iron subsystem and are accompanied by a reorientation of the magnetic moments of the sublattices of the Nd^{3+} and Dy^{3+} ions along the field direction **H**||**c**.
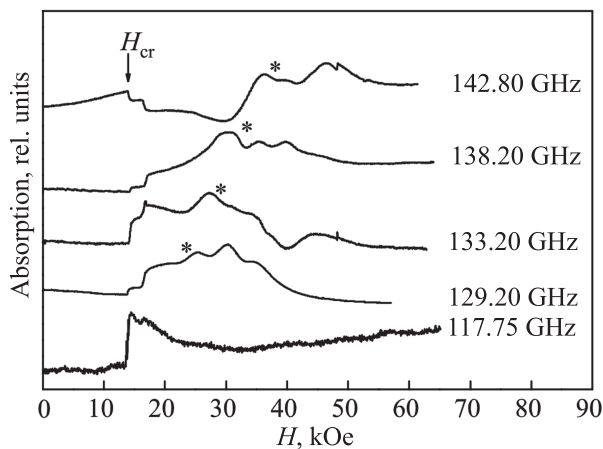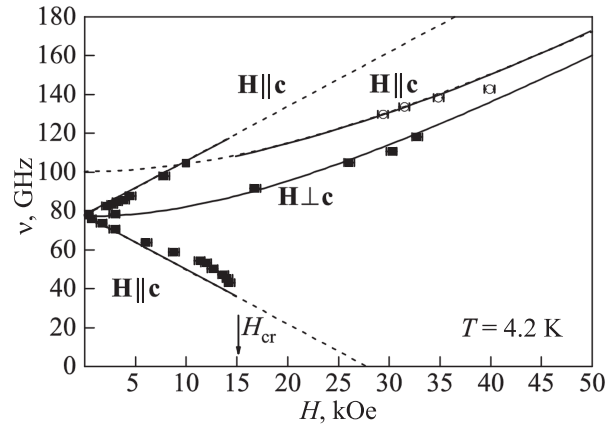

FIG. 4. The AFMR spectrum of crystalline $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ for the easy-plane magnetic structure (**H**||**c**, T = 4.2 K). The asterisks denote the peaks for H > 16 kOe used to construct the frequency-field diagram in Fig. 5.

A calculation of the magnetic phases[17] and the experimental data confirm the appearance of an intermediate state between the collinear and flop phases that takes place in two stages. The first jump is associated with a deviation of the magnetic moments of the iron from the **c** axis by an angle on the order of 25°–30°; a noncollinear antiferromagnetic phase is realized here. The second jump is related to a reorientation of the iron subsystem from the intermediate phase to the flop phase with reorientation of the magnetic moments of the sublattices of the Nd^{3+} and Dy^{3+} ions.

The reason for the appearance of this intermediate state is the competition between the contributions to the total magnetic anisotropy of the iron and rare-earth subsystems of $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$, since the anisotropy of the iron and neodymium subsystems stabilizes the easy-plane magnetic structure, while the dysprosium subsystem stabilizes the easy-axis structure. For certain values of the magnetic field and temperatures, it is energetically favorable for the system to orient the magnetic moments of the iron at some angle to the **c** axis. The high (20 K) temperature for formation of domains during the first order phase transition and the deviation of the magnetic moments of the iron from the principle magnetic axis by 25°–30° are the reasons for the broadening of the AFMR resonance lines.

At 26 K (as the temperature varies from 4.2 K to the Neel point), there is shift in the magnetic state for zero field and an easy-plane magnetic structure of the $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ crystal develops.[9–14] AFMR spectra for external field orientations **H** ∥ **c** and **H** ⊥ **c** are shown in
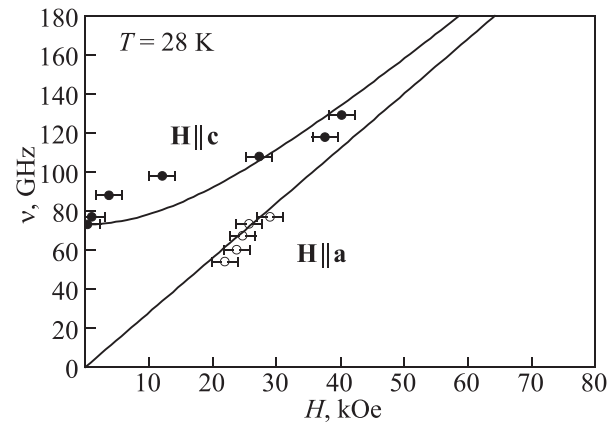

FIG. 7. Frequency-field plots of the AFMR spectra for the easy-axis magnetic structure of the compound $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ with **H** ∥ **c** and **H** ∥ **a**, T = 28 K.

Fig. 6. For a magnet with "easy-plane" anisotropy, when the external magnetic field is oriented along the **c**(**H** ⊥ **l**) axis, only one antiferromagnetic mode with a gap of ∼73.15 GHz is observed at 28 K; with increasing frequency it depends quadratically on the magnetic field. The magnitude of the anisotropy that forms this gap is 0.63 kOe. For a magnetic field **H** ∥ **l** lying in the basis plane, a quasiferromagnetic mode is observed which depends linearly on the magnetic field H_0 (Fig. 7).

The frequency-field curves for the AFMR spectrum at 28 K behave qualitatively the same as those at 4.2 K, but have different magnon excitation energies and are typical of an "easy plane" magnetic structure. Therefore, our resonance measurements are consistent with the data of Ref. 12.

We now examine the resonance properties of crystalline $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ in the paramagnetic region. A broad ($\Delta H = 8.5$ kOe) single absorption line in the EPR spectrum of the Fe^{3+} ions with a g-factor of 2.40 is observed at temperatures of 38–40 K ($T_N = 32$ K) for an external magnetic field oriented along the **c** axis. Further increases in temperature lead to a narrowing of the absorption line with a decrease in the g-factor: $g_c = 2.31$ for $T = 50$ K. Only at temperatures of 77–90 K does g_c reach 2.00. The shape of the EPR spectrum at different temperatures is shown in Fig. 8. Within this temperature range, the EPR spectrum was

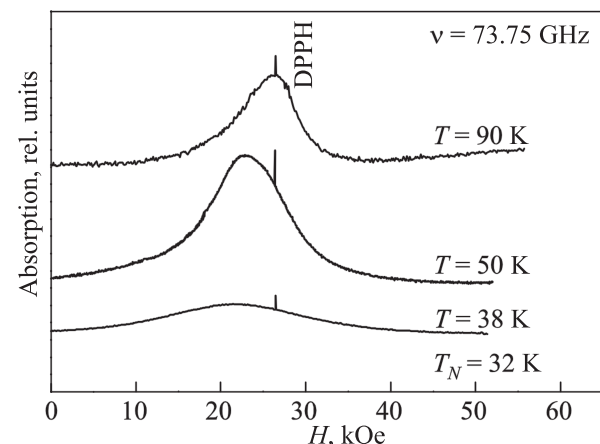FIG. 6. AFMR spectra of crystalline $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ for T = 28 K: **H** ∥ **c** (a) and **H** ⊥ **c**, (b).

FIG. 8. EPR spectra of crystalline $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ for temperatures of 38, 50, and 90 K with **H** ∥ **c**.
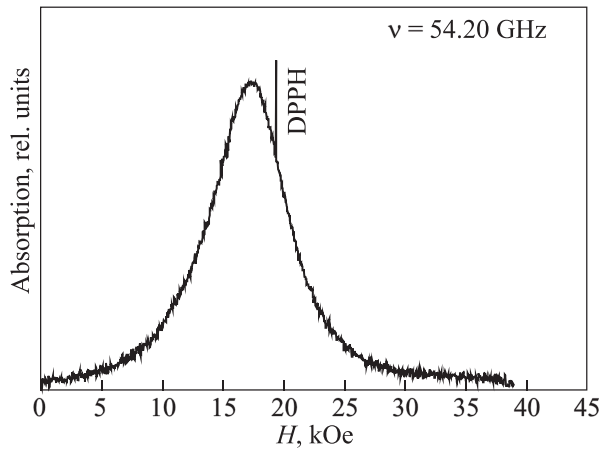
FIG. 9. EPR spectrum of crystalline $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ at $T = 50$ K with $\mathbf{H} \perp \mathbf{c}$.

measured at frequencies of 73.75 and 129.2 GHz, for which the g-factors were the same.

When the external magnetic field is oriented along the **a** axis, however, at 50 K the width of the EPR absorption line and the g-factor are considerably smaller than for $\mathbf{H} \parallel \mathbf{c}$. An absorption resonance line of width $\Delta H \sim 4$ kOe with $g = 2.21$ was observed (Fig. 9), while in pure $NdFe_3(BO_3)_4$ at temperatures of 50–60 K the absorption line of the Fe^{3+} ions corresponded to $g = 2.00$ (Ref. 15) (see Fig. 10). Thus, within a single temperature range, but for different magnetic axes, Fe^{3+} ion EPR absorption lines have been observed with different values of the g factor, $g_c = 2.31$ and $g_a = 2.21$, which both differ from the pure spin state of Fe^{3+}.

The reason for this inconsistency appears to be the admixture of Dy^{3+} ions in the $NdFe_3(BO_3)_4$, since the contributions of the Dy subsystem to the magnetization in the basis plane and along the principal axis of the crystal will be different. Because a unit cell of $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ includes magnetic ions of Fe^{3+}, Nd^{3+}, and Dy^{3+}, it is natural to assume that the magnetic and dynamic properties will be determined by Fe–Fe and Fe–R interactions. In the paramagnetic region the Nd–Nd and Dy–Dy interactions can be neglected. Because of polarization, the influence of Dy^{3+} (which has a small ion radius but a very large magnetic moment) on the oscillations of the Fe^{3+}-ion subsystem will reduce to creating an additional static internal field which,
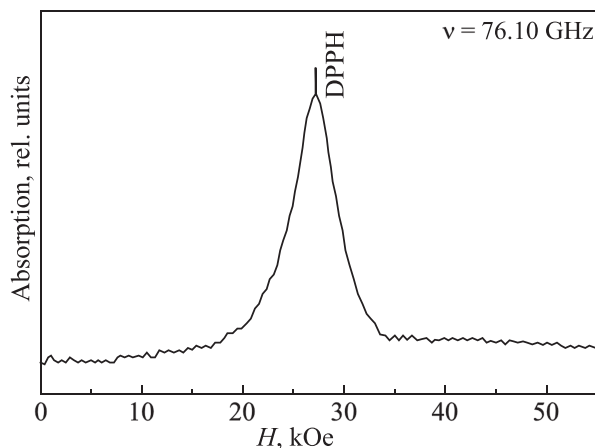
together with the anisotropy field, determines the dynamics of the iron subsystem. In addition, the Fe^{3+} ion has a pure spin state with $g = 2.00$, so that the experimentally observed shifts in the resonance lines of iron can be used to evaluate the effective internal fields of the magnetic interactions created by Dy^{3+} ions along the directions $\mathbf{H} \parallel \mathbf{c}$ and $\mathbf{H} \perp \mathbf{c}$.

In summary, we have studied the effect of replacing Nd^{3+} ions by Dy^{3+} ions on the resonance properties of $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ in the ordered and paramagnetic states. The AFMR technique has been used to detect and study magnetic spin-reordering phase transitions and the frequency-field dependences of the AFMR spectrum owing to the substitution. We have shown that in the resonance measurements, an external magnetic field induces an "easy axis"-"easy plane" spin-reorienation phase transition in the form of a two-step anomaly. This anomaly is related to the existence of an intermediate state (noncollinear antiferromagnetic phase) between the collinear and spin-flop phases at fields of $H_1 = 14.920$ kOe and $H_2 = 15.850$ kOe at 4.2 K. The magnetic phase transition is a consequence of competition among Nd–Fe and Dy–Fe exchange interactions. The reduction in the fields at which the transitions take place with increasing temperature indicates an instability in the collinear phase, while the parallel susceptibility increases.

For an external magnetic field oriented along the **c** axis at 4.2 k, the frequency-field dependences for the AFMR spectrum in fields up to 15 kOe correspond to a uniaxial magnetic structure with a gap of 77.2 GHz in the spin excitation spectrum, and in fields above 16 kOe these correspond to an easy-plane structure with an energy gap of 100.3 GHz. At high temperatures (28 K), where there is no phase transition with respect to the magnetic field, the AFMR spectrum ($\mathbf{H} \perp \mathbf{l}$, $\mathbf{H} \parallel \mathbf{l}$) and its frequency dependence are typical of a magnetic structure with an "easy plane" anisotropy. The magnitudes of the magnon excitation gaps for the compound $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ are caused by the combined action of the anisotropies of the iron and rare-earth subsystems, for both the easy axis and the easy plane magnetic structures.

In the paramagnetic region, because of polarization, the effect of added Dy^{3+} ions on the Fe subsystem of crystalline $Nd_{0.75}Dy_{0.25}Fe_3(BO_3)_4$ leads to the appearance of an additional static internal field that shifts the resonance field of the EPR absorption line of the Fe^{3+} ions and shows up as a change in the g-factor over a wide range of temperatures. A purely resonance line of the Fe^{3+} ions with $g = 2.00$ observed only at temperatures on the order of 90 K.

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FIG. 10. EPR absorption line of crystalline $NdFe_3(BO_3)_4$ at $T = 60$ K with $\mathbf{H} \parallel \mathbf{c}$.[15]

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