# Magnetic anomaly of the Mössbauer line intensity for $Fe^{57}$ nuclei in iron garnets

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It is shown that the temperature dependence of the probability of the Mössbauer effect for $Fe^{57}$ nuclei in $Y_3Fe_5O_{12}$ and $Tb_3Fe_5O_{12}$ iron garnets exhibits a magnetic-transition anomaly due to "exchange perturbation" of the phonon spectrum. The nature of the anomaly is such that the effective Debye temperature increases on going from the paramagnetic temperature region to the region $T < T_c$ ($\Delta\Theta_D/\Theta_D \approx 25\%$ at $T = 293°K$). The anomaly itself is practically entirely due to the strong dependence of the energy of the exchange interaction between the iron sublattices on the interatomic distance.

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## 1. INTRODUCTION

At present there are only several experimental papers on the magnetic anomalies in the Mössbauer effect,[1–6] and it is shown there that the anomaly observed in the area $S$ and the shift $\delta$ of the Mössbauer spectrum is due to a magnetic transition that alters the phonon spectrum of the crystal. This is why the problem of magnetic anomalies in the Mössbauer effect has remained so far one of the practically uninvestigated regions of solid-state physics. Yet the importance of investigating these anomalies is quite obvious. Their very existence uncovers new possibilities for the study of magnon-phonon interactions in solids.

The main cause of the magnetic anomalies of the Mössbauer-spectra parameters is that the exchange energy $W_{exch}$ depends on the interatomic distances $a$.[7] Because of this dependence, the change of the magnetic state of the material (say on going from the parametric temperature region into the region where magnetic order exists) leads to a corresponding "exchange perturbation" of the elastic constants. Within the framework of a rigorous calculation, such a transition from one state to another leads to the need for renormalization of the phonon spectra.

Obviously, the magnetic anomalies should generally speaking be larger the stronger the dependence of $W_{exch}$ on $a$. Accordingly, the criteria used to seek research objects for which sufficiently large magnetic anomalies are to be expected can be a strong dependence of the temperature $T_C$ of the magnetic transition on the pressure $P$, a large value of the magnetostriction, as well as magnetic anomalies of the elastic moduli.[7]

Among the crystals for which anomalies of the temperature dependence of the Lamb–Mössbauer factor $f(T)$ can be expected are, in particular, iron garnets (which contain the Mössbauer isotope $Fe^{57}$, which is convenient for the investigations). According to the available data, the exchange energy in iron garnets depends relatively strongly on the interatomic distances. This can be assessed from the $T_C(P)$ dependence. Thus, for $Y_3Fe_5O_{12}$ the value reached by $\partial T_C/\partial P$ is 0.7 °K/kbar.[8]

The magnetic properties of ferrimagnetic oxides with garnet structure have a number of distinguishing features. Their magnetic structure can be represented in the form of three sublattices whose magnetization vectors are collinear. The exchange energy of the iron garnet is determined mainly by the negative interaction of two iron sublattices $a$ and $d$, owing to which their magnetizations are established antiparallel to one another, with $M^d > M^a$. The third sublattice, $c$, consisting of rare-earth (or Y) atoms, is characterized by a relatively weak intrasublattice interaction. Its magnetization vector $\mathbf{M}^c$ is antiparallel to the resultant vector $\mathbf{M}^a + \mathbf{M}^d$.[9]

The dynamic properties of the Mössbauer nuclei $Fe^{57}$ in iron garnet have been investigated very little. The available experimental results do not present a clear picture either of the character of the temperature dependence of the probability of the effect or of its correlation with the magnetic properties.

Table 1 lists data on the factor $f$ and the effective Debye temperature $\Theta_D$ for $Fe^{57}$ nuclei in iron garnets (including substituted ones), taken from those few papers in which the authors have analyzed the experimental data on the temperature dependence of the area $S$ of

TABLE 1.

| Compound | Data on $\Theta_D$, $f$, or $S$ | References |
|---|---|---|
| $Y_3Fe_5O_{12}$ | $\Theta_{D^d} > \Theta_{D^a}$ | [10] |
| $Y_3Fe_5O_{12}$ | $f^d/f^a \approx 1.1$ ($T > T_C$) | [11] |
| $Y_3Fe_5O_{12}$ | $f^a/f^d \approx 0.94$ ($T = 293$ K)<br>$S^d/S^a = 1.55 \div 1.70$<br>(293 $K < T <$ 780 K)<br>$\Theta_D{}^d = 406 \pm 15$ K<br>$\Theta_D{}^a = 306 \pm 15$ K | [12] |
| $Ca_3Fe_3Si_3O_{12}$ | $\Theta_D{}^a \approx 400$ K | [13] |
| $Ca_3Fe_3Ge_3O_{12}$ | $\Theta_D{}^a \approx 400$ K | [13] |
| $YCa_2Sn_2Fe_3O_{12}$ | $\Theta_D{}^d \approx 550$ K | [14] |
| $CdCa_2Sn_2Fe_3O_{12}$ | $\Theta_D{}^d \approx 550$ K | [14] |
| $Y_3Fe_5O_{12}$ | $\Theta_D{}^a \approx 378$ K<br>$\Theta_D{}^d \approx 343$ K<br>($T > T_C$) | [15] |
| $Y_3Fe_4AlO_{12}$ * | $\Theta_D{}^a \approx 467$ K<br>$\Theta_D{}^d \approx 405$ K<br>($T > T_C$) | [16] |
| $Y_3Fe_5O_{12}$ * | $\Theta_D{}^a \approx 378$ K<br>$\Theta_D{}^d \approx 343$ K<br>($T > T_C$) | [16] |

*The authors of [16] have observed in these compounds an appreciable anharmonicity at $T > T_C$.

the Mössbauer spectra. It is seen from the table that the data of [10-14] are qualitatively in agreement with one another: according to the estimates, $\Theta_D^d$ is somewhat larger than $\Theta_D^a$. As to Alekseev *et al.*,[15] their results contradict those of others. In view of the purpose of our investigation, attention is called also to the fact that the authors of [16] have concluded, as a result of reducing their data on the $S(T)$ dependence, that the factor $f$ has a magnetic anomaly that corresponds to an appreciable change of the effective Debye temperature $\Delta\Theta_D = \Theta_D(T<T_C) - \Theta_D(T>T_C) < 0$. The authors of the other papers did not notice this anomaly (and apparently did not search for it). The question of the singularities of the dynamic properties of iron nuclei in iron garnet calls for further study.

In this paper we report the results of our investigations of the temperature dependence of the intensity of the Mössbauer lines for $Fe^{57}$ nuclei in the iron garnet $Y_3Fe_5O_{12}$ and $Tb_3Fe_5O_{12}$. From the point of view of the problem of magnetic anomalies in the Mössbauer effect, the most important difference between the chosen research objects is that the sublattice $c$ becomes nonmagnetic when the $Tb^{3+}$ ions are replaced by $Y^{3+}$.

## 2. SAMPLES. REDUCTION OF SPECTRA

The iron-garnet samples were prepared in accordance with the usual ceramic technology. The initial components were the oxides $Y_2O_3$, $Tb_4O_7$, and $\alpha$-$Fe_2O_3$ [of ChDA (analytically pure) grade]. The iron in $\alpha$-$Fe_2O_3$ was enriched with $Fe^{57}$ to 20%.

According to the x-ray structure analysis data, the samples were single-phase. The temperature dependence of the magnetizations of the samples agreed with the known data.[9] The thicknesses of the absorbers were 0.125, 0.208, 0.287, and 0.451 mg $Fe^{57}/cm^2$ for $Tb_3Fe_5O_{12}$ and 0.0776, 0.133, 0.196, 0.255, and 0.348 mg $Fe^{57}/cm^2$ for $Y_3Fe_5O_{12}$.

The Mössbauer spectra were computer-reduced by a program described in [17], the distinguishing feature of which is the possibility of varying, in a wide range, the model assumptions within the framework of which the spectra are interpreted.

In the reduction of the Mössbauer spectra it was assumed, in accordance with Table 1, that $\Theta_D^d \neq \Theta_D^a$. It was assumed also that the spectra of the investigated iron garnets are superpositions of two Zeeman sextets corresponding to the sublattices $a$ and $d$ (in analogy with the procedure used in [10,15,16].) By the same token, it was assumed that the $a$ sublattice contains Fe atoms that occupy positions that are magnetically equivalent. Generally speaking, a more consistent reduction of the spectrum for the $a$ sites would take into account the presence of two types of positions in the $a$ sublattices, with different orientations of the principal axes of the electric field gradient tensor relative to the field $\mathbf{H}_n$. Since the principal aim of our investigation was an attempt to observe a magnetic anomaly in the dependence of the spectrum area $S(T)$, this circumstance can be disregarded, bearing in mind the fact that the values of $H_n$ are the same (accurate to the contribution from the dipole-dipole interaction, which does not exceed 6 kOe[18]) for all the Fe nuclei in the $a$ sites, and the relatively small value of the quadrupole displacement $\varepsilon$ of the components of the hyperfine magnetic structure ($\varepsilon \lesssim 0.25$ mm/sec).

To determine the probability $f$ of the effect we used the area method.[19] It is known that this method is most advantageous when it comes to determining the probability of the radiation-source effect $f_s$ (see, e.g.,[1]), for the area $S$ of the spectrum and the probability $f$ are connected in this case by the obvious relation $S = \text{const} \cdot f_s$. The constant can be determined here from experiments with a standard sample. As to the probability $f_a$ of the effect for the absorber, it cannot be determined so simply, since

$$S/f_a \neq \text{const} \tag{1}$$

in view of the "saturation effect." It can be shown, however, following [19], that also in the case of an absorber a successive application of the area method makes it possible to determine, by suitable reduction of the data, the value of $f_a$.

In analogy with [19], we described the area of the spectrum by the function

$$S = f_s \frac{c}{E_0} \int_{-\infty}^{\infty} \left(1 - \exp\left\{-\sum_i n_a^i \sigma^i(E) f_a^i\right\}\right) dE, \tag{2}$$

where $c$ is the speed of light, $E_0$ is the energy of the $\gamma$ transition, $n_a^i$ is the number of Mössbauer nuclei in the sublattices with number $i$ per unit area of absorber, and $\sigma^i(E)$ is the cross section for the resonant absorption of the $\gamma$ quanta with energy $E$.

In accordance with the idea used in [20], we take into account the broadening of the components of the hyperfine structure of the absorber line, writing down the function $\sigma^i(E)$ in the form

$$\sigma^i(E) = \sigma_0 \sum_{k=1}^{p^i} \frac{\Gamma}{(\Gamma_k^i)_a} \frac{\beta_k^i}{1+\xi_k^i}, \tag{3}$$

where

$$\sigma_0 = \frac{2I_e+1}{2I_g+1} \frac{\lambda^2}{2\pi} \frac{\Gamma_\gamma}{\Gamma}, \quad \xi_k^i = \left(\frac{E - E_0 - E_0 v_k^i/c}{(\Gamma_k^i)_a/c}\right)^2, \tag{4}$$

$I_e$ and $I_g$ are the spins of the nuclei in the excited and in the ground states, $\lambda$ is the wavelength of the resonant radiation, $\Gamma_\gamma$ and $\Gamma$ are the radiative and total widths of the level, $p^i$ is the number of the components of the hyperfine structure of the $i$-th partial spectrum, $v_k^i$ and $(\Gamma_k^i)_a$ are the Doppler shift and the width of the $k$-th component. The relative intensities $\beta_k^i$ obey the normalization condition

$$\sum_{k=1}^{p^i} \beta_k^i = 1.$$

Formula (2) together with (3) and (4) make it possible not only to specify concretely the character of the cor-

relation of the values of $S$ and $f_a$, described with the aid of the inequality (1), but also to take into account the saturation effect.

The importance of taking into account the saturation effect is illustrated by Fig. 1. It is seen, in particular that the abrupt change of the character of the $S(T)$ dependence near $T_C$, obtained for a "thick" sample, can be mistaken for a magnetic anomaly due to the influence of the exchange interaction on the phonon spectrum of the crystal. The latter anomaly, however, is characterized by the fact that it manifests itself in a very narrow temperature interval (on the order of 1–2 °K at $T_C \approx 300$ °K, at $H_n(0) \approx 500$–300 kOe, and at the usual character of the dependence of $H_n$ on $T$). In addition, it always corresponds to a seeming change $\Delta\Theta_D > 0$, which can facilitate the analysis of the experimental data[4] in a number of cases (especially in the presence of a true change $\Delta\Theta_D < 0$).

We note that at sufficiently high values of $\varepsilon$ and $H_n$, that part of the saturation effect which is due to the hyperfine interactions is "lifted." According to calculations, for samples $n_a \lesssim 0.1$ mg $Fe^{57}/cm^2$ thick, this takes place at $\varepsilon > 2\Gamma \approx 0.2$ mm/sec and $H_n \gtrsim 10\Gamma \approx 30$ kOe.

## 3. RESULTS OF THE REDUCTION OF THE SPECTRA

The saturation effect was taken into account in experiments with a series of samples of varying thickness in the paramagnetic temperature regions, where the correction for this effect is particularly large. According to preliminary spectrum interpretations, the ratio $S^d/S^a$ of the areas of the partial spectra differed only insignificantly from the sublattice population ratio $N^d_{Fe}/N^a_{Fe} = 1.5$. For this reason, when account was taken of the saturation effect, it was assumed that $\Theta^d_D = \Theta^a_D = \Theta_D$. This assumption is justified also by the purpose of the investigation: inasmuch as in iron garnets the dominant process is exchange interaction between the $a$ and

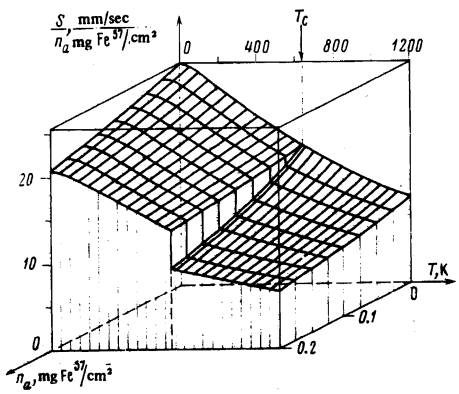


FIG. 1. Saturation effect in the absence of a magnetic anomaly of the Lamb-Mössbauer factor. The family of curves was calculated from formulas (2), (3), and (4) at $f_s = 0.74$ ($Co^{57}$ in Cr), $\Gamma_a = \Gamma$, $\varepsilon = 0$, $\Theta_D = 400$ °K under the assumption that the temperature dependence of the magnetic field $H_n$ is given by $H_n = D(1 - T/T_C)^\beta$ with $T_C = 645$ K, $D = 210$ Γ and $\beta = 0.33$ (orthoferrite case[21]).

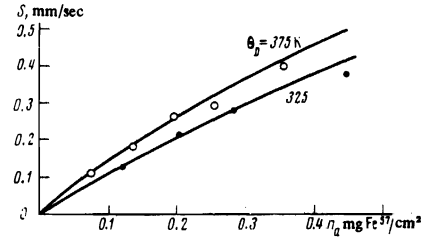


FIG. 2. Dependence of the area of the spectrum on the sample thickness at $T = 583$ °K: o—for $Y_3Fe_5O_{12}$, ●—for $Tb_3Fe_5O_{12}$.

$d$ sublattices, the exchange corrections $\Delta\Theta^a_D$ and $\Delta\Theta^d_D$ should be of the same sign. It is important also that data by others also yield relatively small differences between $\Theta^a_D$ and $\Theta^d_D$.

The reduction of the data on the dependence of the spectrum area $S$ on the sample thickness $n_a$ (Fig. 2) has enabled us to establish that at $T > T_C$ the effective Debye temperature $\Theta_D$ is 375 ± 15 °K and 325 ± 15 °K for $Y_3Fe_5O_{12}$ and $Tb_3Fe_5O_{12}$, respectively. We note that the value of the parameter $\Theta_D$ for the iron garnet $Y_3Fe_5O_{12}$, for which it becomes possible to carry out a comparison with data by others, agrees well with the results of[12,15] (see Table 1).

Taking into account the obtained values of $\Theta_D$, we have calculated the functions $S(T)$ for the samples of both iron garnets under the assumption that exchange interactions do not exert any influence on the vibrational spectrum of the iron nuclei. We used in the calculations formulas (2)–(4) as well as data on the temperature dependences of the fields $H^a_n$ and $H^d_n$ (Fig. 3).

The calculated plots of $S(T)$ are shown in Fig. 4 together with the experimental values of $S$ at different values of $T$.

## 4. DISCUSSION

As seen from Fig. 4, at $T < T_C$ the experimental $S(T)$ plots differ substantially from those calculations under the assumption of constant $\Theta_D$. The character of the discrepancy indicates that the transition from the paramagnetic temperature region to temperatures $T < T_C$ is accompanied by a change in the effective Debye temperature $\Delta\Theta_D > 0$. A tentative calculation based on the use

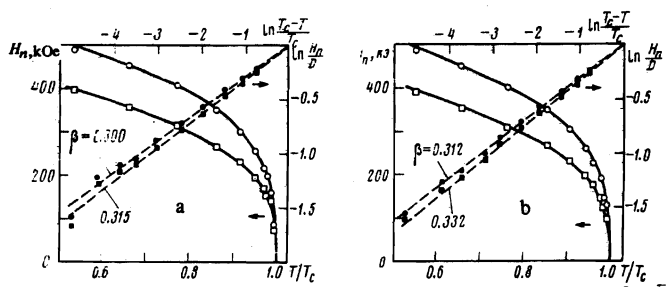


FIG. 3. Temperature dependence of the fields $H^a_n$ (o) and $H^d_n$ (□): a) at the $Fe^{57}$ nuclei in $Y_3Fe_5O_{12}$, b) at the $Fe^{57}$ nuclei in $Tb_3Fe_5O_{12}$.

TABLE 2.

| No | Substance | Crystal structure | $T_C$, K | Magnetic transition | Mössbauer isotope | Function exhibiting anomaly | Allowance for saturation | References |
|---|---|---|---|---|---|---|---|---|
| 1 | $MnAu_2$ | Tetragonal | 378 | AF → P<br>AF → F | $Fe^{57m}$ | $f(T)$<br>$f(H)$ | No need | [1] |
| 2 | $Mn_2Sn$ | Hexagonal | 260 | FI → P | $Sn^{119}$ | $S(T)$ | ? | [2] |
| 3 | $FeF_3$ | Rhombic | 370 | WF → F | $Fe^{57}$ | $\delta_T(T)$ | — | [3] |
| | | Cubic | 485 | F → P | $Fe^{57}$ | $S(T)$ | Yes | [4] |
| 4 | Fe – Ni Invar alloy | | | | | | | |
| 5 | $FeSn_2$ | Tetragonal | 380 | AF → P | $Fe^{57}$ | $f(T)$ | Yes | [5] |
| 6 | Pd-Co alloy | Cubic | | | $Sn^{119}$ | $S(T)$ | Yes | [6] |
| 7 | $HoFeO_3$ | Perovskite | 640 | WF → P | $Fe^{57}$ | $S(T)$, $\delta_T(T)$ | Not accounted for | [22] |
| 8 | $Y_3Fe_5O_{12}$ | Garnet | 553 | FI → P | $Fe^{57}$ | $S(T)$ | Yes | Present work |
| 9 | $Tb_3Fe_5O_{12}$ | Garnet | 559 | FI → P | $Fe^{57}$ | $S(T)$ | Yes | Present work |

*Note.* AF—antiferromagnetism, P—paramagnetism, F—ferromagnetism, FI—ferrimagnetism, WF—weak ferromagnetism.

of formulas (2)–(4) at $\Theta_D \neq$ const yields for room temperatures $\Delta\Theta_D(Y_3Fe_5O_{12}) \approx 100$ °K and $\Delta\Theta_D(Tb_3Fe_5O_{12}) \approx 85$ °K.

The difference between the values of $\Theta_D$ obtained by us for $Y_3Fe_5O_{12}$ and $Tb_3Fe_5O_{12}$ offers evidence of the dependence of the vibrational spectrum of the iron nuclei not only on the nearest environment, but also on the second and succeeding coordination spheres.[1)]

At the same time, the data on the exchange corrections $\Delta\Theta_D/\Theta_D$ for the two investigated iron garnets point to a negligibly small contribution of the rare-earth sublattice to the magnetic anomaly of the intense Mössbauer lines, namely, replacement of the magnetoactive ions $Tb^{3+}$ by the nonmagnetic $Y^{3+}$ does not lead to a significant change in the character of the $S(T)$ dependence (and of the correction $\Delta\Theta_D/\Theta_D$). This result indicates that in the $S(T)$ plots of other iron garnets we should also expect magnetic anomalies that lend themselves fully to measurement.

In connection with these results, we wish to call attention to the fact that the magnetic anomalies of the Mössbauer-spectrum parameters, due to the influence of exchange interactions on the phonon spectrum, turn out to be quite typical of magnetic crystals. As seen from Table 2, the substances for which these anomalies have already been observed include objects that are quite dissimilar from the point of view of the magnetic structure, such as the ferrimagnet $Mn_2Sn$ and the helicoidal antiferromagnet $MnAu_2$, the Invar alloy Fe–Ni, alloy Pd–Co, the weak antiferromagnet $FeF_3$, the antiferromagnet $FeSn_2$, and the iron garnets $Y_3Fe_5O_{12}$ and $Tb_3Fe_5O_{12}$.

Table 2 does not include the results of studies in which anomalies of the shift $\delta$, which are caused by the change of the isomer shift (and not by the temperature shift $\delta_T$), as well as debatable cases. Among the latter were studies in which the observed magnetic anomaly of the temperature dependence of the line shift $\delta(T)$ could be ascribed both to an anomaly in the dependence of the isomer shift proper $\delta_I(T)$ and of the dependence of the relativistic correction $\delta_T(T)$. This is the situation, for example, with the compounds FeSn,[23] FeGe,[24] $FeCl_3$,[25] $V_2O_3$,[26] and $\alpha$-$Fe_2O_3$.[27]

We note also that in most studies in which magnetic anomalies of the Mössbauer-spectrum parameters were observed, there was no mention at all of the saturation effect. Without invoking additional data, the question of the nature of the anomalies observed in these studies (see Table 2) cannot be regarded as finally explained. Worthy of attention in this connection are the compounds $Mn_2Sn$ and $HoFeO_3$, for which the character of the change of the functions $S(T)$ is the same as in the ordinary saturation effect (Fig. 1).

With the iron garnets investigated by us as examples, it is seen that the study of the magnetic anomalies of the Mössbauer-spectrum parameters encounters a large number of methodological difficulties, the principal among which is due to the need for taking the saturation effect into account. This explains possibly why the magnetic anomalies in the Mössbauer effect have been observed so far only for a few magnetic crystals.

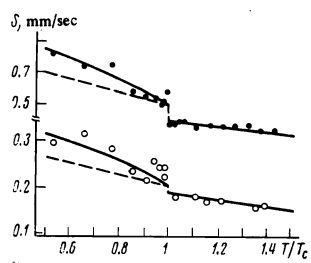


FIG. 4. Area of the spectrum as a function of the temperature: o—for $Y_3Fe_5O_{12}$ at $n_a = 0.133$ mg $Fe^{57}/cm^2$ and ●—for $Tb_3Fe_5O_{12}$ at $n_a = 0.451$ mg $Fe^{57}/cm^2$. The dashed curves correspond to the theoretical calculations under the assumption of constant $\Theta_D$.

[1)]A similar conclusion can be arrived at also as a result of comparing the data of[12] and[14]: for example, on going from $Y_3Fe_5O_{12}$ to the substituted iron garnet $YCa_2Sn_2Fe_3O_{12}$, the parameter $\Theta_b^d$ increases 406 °K[12] to 550 °K[14].


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