Investigation of the properties of the dislocation EPR spectra in silicon

S. V. Broude, V. A. Grazhulis, V. V. Kveder, and Yu. A. Osip'y an

Institute of Solid State Physics, USSR Academy of Sciences
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We investigated the properties of the dislocation EPR spectra in Si in the temperature interval from 1.3 to 150 °K. At helium temperatures we observed anomalies in the behavior of the dispersion signals Χ under conditions of adiabatic rapid passage (ARP) through resonance. It is shown that the spectrum of the D centers has a hyperfine (hf) structure, with a linewidth ΔH₁ = 0.2–0.3 Oe (the distance between neighboring hf lines is of the order of their width). It is established that under ARP conditions excitations are transferred between the hf lines as a result of spin-spin interactions with a characteristic time τ₂ equal to 3–10 sec in the range 1.3–4.2 °K and weakly dependent on the temperature and on the microwave power. We measured the dependence of the integrated intensity of the absorption signals Χ on the temperature in the 20–150 °K range. A strong deviation from the Curie law was observed at T = 40–50 °K. The temperature dependence of the quantity τ₂ was measured in the same temperature range, under the assumption that the hf lines have a Lorentz shape. An anomaly at T = 40–50 °K was observed also on the plot of τ₂ = 1/ΔH₁. It is concluded that a magnetic phase transition takes place in the D-center system at 40–50 °K, and consequently the dislocations in Si can be regarded as models of one-dimensional chains of spins with exchange interactions.

INTRODUCTION

In earlier investigations(1–3) we observed the EPR spectrum of hitherto unknown centers (called D centers) produced when silicon single crystals are plastically deformed, and investigated certain properties of this spectrum. The results were explained under the assumption that the D-center EPR signal is due to broken bonds in the cores of dislocations with edge components. The observed spectrum turned out to be anisotropic and had an incompletely resolved structure with line width 2–5 Oe; the number of the lines and their width were independent of the temperature in the interval 1.3–300 °K. The effective g factors of the line ranged from 2.00 to 2.01 (all the measurements of(1–3) were made in the 3-cm band). It was also observed (by the EPR method) that the dislocations exert acceptor action in single crystals of silicon doped with phosphorous, and the filling factor of the “dangling” bonds by the captured electrons was determined.

Electron microscopy of the samples has shown that the dislocations form a complicated system of intersecting lines with a weakly pronounced anisotropy in the spatial orientation. The dislocation tangles can lead to significant interactions between spins localized on various dislocations, and accordingly complicate the study of the “individual” properties of the dislocation centers. At the same time, it is a rather complicated task to obtain at present a homogeneous distribution of linear dislocation at a density ~10¹⁵ cm⁻². It must be assumed, however, that the principal interaction is still that between the spins localized within a single dislocation, inasmuch as the distance between the neighboring spins in the dislocation core (in the case of an edge or 60° dislocation) is comparable with the lattice constant, it follows that the properties of the D centers should be determined primarily precisely by these interactions. Since the distance between neighboring spins is small, an exchange interaction may take place, which usually leads to a bowing of the spectral lines in disordered systems and, generally speaking, can cause spontaneous ordering of the spins, including also in quasi-one-dimensional systems such as dislocations(3,4).

Our earlier investigations(1–3) have not made it possible to establish unequivocally whether magnetic ordering actually exists on the dislocations(3,4), and consequently whether the dislocations can be regarded as models of linear chains of exchange-coupled spins. Recognizing that the study of the properties of one-dimensional spin chains, as well as the properties and structure of the dislocation cores is presently of great interest, we have undertaken further investigations of the properties of the D-center spectra in a wide range of temperatures.

SAMPLES AND EXPERIMENTAL PROCEDURE

We investigated silicon single crystals with not more than 10¹⁴–10¹⁵ cm⁻³ of electrically active impurities. The single crystals were grown by crucibleless melting in vacuum. The samples were parallelepipeds whose edges coincided with the directions [111], [112], [110] and measured 3.5 × 4 × 9 mm. The samples were deformed by bilateral compression along the [110] direction in an argon atmosphere at 700 °C. The sample preparation procedure is described in greater detail in(1,2). The degree of sample deformation reached 4–5%.

The measurements were made by placing the samples in the center of a rectangular gold-plated resonator operating in the H₁₀₀ mode. The resonator Q was 3000–4000 and varied little with temperature. To determine the g factors of the lines, we registered simultaneously with the investigated spectrum the signal from the Mn⁺⁺ ions in MgO powder, introduced into the interior of the quartz holder for the sample. The EPR signals were observed with a bridge-type superheterodyne spectrometer at a microwave field frequency ~9300 MHz in the temperature range 150–1.3 °K. The measurements were made with the static magnetic field modulated at 800 Hz, followed by synchronous detection at this frequency. The modulation amplitude ranged from 0.03 to 20 Oe. For the measurements at 4.2 to 77 °K, the temperature was regulated by blowing helium gas of the required temperature through the cryostat in which the resonator was placed. The sample tempera-
ture was measured with an Au-eu thermocouple and was maintained constant by controlling the power used to heat the helium gas in the cryostat from $T = 4.2^{\circ}K$ to the required value. The temperature was maintained in this interval within not more than 0.5°K. The temperature in the 4.2–1.3°K range was regulated by pumping off the vapor of the liquid helium which was poured in the cryostat and in which the resonator was immersed.

At temperatures 4.2–1.3°K, in view of the large spin-lattice relaxation time, the signal was recorded with adiabatic fast passage (AFP) through the resonance. The AFP conditions call for satisfaction of the following inequalities:

$$\omega_M T_1 > 1, \quad \gamma H_1 > \gamma H_0 \omega_M, \quad \gamma H_1 T_1 T_2 > 1,$$

where $\omega_M$ and $H_M$ are the frequency and amplitude of the modulation of the "static" field $H_0$, $H_1$ is the amplitude of the microwave field at the sample, $\gamma$ is the gyromagnetic ratio, $T_1$ and $T_2$ are the times of the spin-lattice and spin-spin relaxation. At helium temperatures, the recorded signal of the real part $\chi'$ of the susceptibility was 50–100 times larger than the absorption signal $\chi''$.

At higher temperatures, the measurements were made under conditions of slow passage through resonance, when

$$\frac{dH_t}{dt} > T_1, \quad T_2,$$

where $\Delta H_t$ is the width of the resonance line and $dH/dt$ is the rate of change of the "static" field. In this case the signal was the derivative of the dispersion or absorption signals.

We present below the results of an investigation of the structure of the EPR spectrum of the D centers as determined from the effects of passage through resonance at helium temperatures, and also investigations of the temperature dependence of the spectrum intensity at $T = 20–150^{\circ}K$.

**EXPERIMENTAL RESULTS AND DISCUSSION**

1. **Investigation of the Spectrum Structure**

Figure 1 shows plots of the signal $\chi'$ for D centers in AFP regime. The regimes under which the spectra were recorded differ only in the direction of the scanning of the magnetic field $H_0$, as indicated by the arrows. The redistribution of the spectral line intensity is observed as a function of the sign of $dH_0/dt$. This phenomenon offers evidence of the presence of an appreciable coupling between the spin groups corresponding to different lines of the spectral structure, i.e., apparently to groups of dislocations with different crystallographic orientations. This coupling, as already noted, can be due to intersections of dislocation lines. This hypothesis is favored also by the fact that the effect varies somewhat in magnitude from sample to sample.

At temperatures $T = 20–150^{\circ}K$, the EPR spectrum of the D centers has a "hyperfine" structure, as observed as a function of the sign of $dH_0/dt$. This spectrum has revealed an "anomalous" behavior of the dispersion signal $\chi'$ in the AFP regime when the magnetic-field scanning is stopped. The experiment was performed in the following manner: The signal $\chi'$ was first recorded without stopping the magnetic-field scanning (Fig. 2a) with the drive of the x-y recorder charge turned on, after which the signal was recorded again at the same field-scanning direction (after a time $T > T_1$), but now the scanning was turned off at the instant of passage through the point A (Fig. 2b) and the recorder plotted the transient of the stationary signal. As seen from Fig. 2b, the signal first decreases abruptly (within <1 sec) after the scanning is turned off, and then it increases gradually to a stationary value. The growth of $\chi'$ is not described by a single exponential, and it is impossible to define unambiguously a time constant for this process. An estimate was made, however, of the time $\tau^*$ required for the signal to grow to the level 0.5 ($A_1 - A_2$). This time turned out to depend little on the temperature and the microwave field $H_1$.

Some figures are listed in the table.

<table>
<thead>
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<th>$T$, °K</th>
<th>$\gamma H_1$, μeV</th>
<th>$H_0$, Oe</th>
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<tr>
<td>4.2</td>
<td>2.7</td>
<td>5.5</td>
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The study of the effects of local saturation of the spectrum has revealed an "anomalous" behavior of the dispersion signal $\chi'$ in the AFP regime when the magnetic-field scanning is stopped. The experiment was performed in the following manner: The signal $\chi'$ was first recorded without stopping the magnetic-field scanning (Fig. 2a) with the drive of the x-y recorder charge turned on, after which the signal was recorded again at the same field-scanning direction (after a time $T > T_1$), but now the scanning was turned off at the instant of passage through the point A (Fig. 2b) and the recorder plotted the transient of the stationary signal. As seen from Fig. 2b, the signal first decreases abruptly (within <1 sec) after the scanning is turned off, and then it increases gradually to a stationary value. The growth of $\chi'$ is not described by a single exponential, and it is impossible to define unambiguously a time constant for this process. An estimate was made, however, of the time $\tau^*$ required for the signal to grow to the level 0.5 ($A_1 - A_2$). This time turned out to depend little on the temperature and the microwave field $H_1$.

Some figures are listed in the table.

If the field scanning is again turned on after the establishment of the stationary signal (point B of Fig. 2), then the signal decreases abruptly (within <1 sec) by a certain amount, and then varies smoothly in agreement with the spectrum shape shown in Fig. 2a.

We measured the signal amplitudes $A_1$, in the case of continuous scanning of the field and the amplitudes of the steady-state signals $A_2$ (after turning the scanning off) as functions of $H_1$ and $H_M$. The results of these measurements are shown in Fig. 3. We note that at $H_1 = 3 \times 10^{-3}$ Oe the AFP condition $\gamma H_1 \gg H_M \omega_M$ is satisfied only for $H_M < 0.1$ Oe, whereas at $H_1 = 2 \times 10^{-3}$ Oe the AFP condition is valid for all the $H_M$ indicated in Fig. 3. As seen for this figure, the functions $A_1 = F(H_M)$ and $A_2 = F(H_M)$ are essentially different. Attention should be called first to the nonmonotonic character of $A_3 = F(H_M)$ at $H_1 = $ const, namely the presence of "oscillations," An appreciable deviation from monotonicity (first maximum) appears at $H_M \sim 0.2–0.3$ Oe, whereas the width of the observed spectral lines was of the order $2–4$ Oe$^{1/2}$.

To explain the results of Fig. 2 and 3, we propose that the D-center spectrum has a "hyperfine" structure,
i.e., it consists of spin packets between which there exist interactions with a certain characteristic time \( \tau_s \approx \tau^* \). We emphasize that the term "hyperfine" structure does not mean that it is connected with the hyperfine interaction of the electron spins with the nuclei of the isotope \( ^{29}\text{Si} \), since we assume that the presence of the "hyperfine" structure may be due in our case to the distribution of the dislocations that produce the EPR signal over the lengths and over the crystallographic directions in the lattice. The fact that \( \tau^* \) depends little on the magnetic field position of the spin packets is that at helium temperatures the spin-lattice relaxation time \( \tau_1 \) varies like \( T^{-1/2} \) suggests that \( \tau_s \) is most likely the characteristic time of certain spin interactions that cause excitations to be transferred between spin packets. We note that this time is macroscopic, namely, \( \tau_s \sim 3-10 \text{ sec} \) in the region 1.3-4.2 K and \( \tau_s \sim \tau_1 \). Since \( \tau_s \) varies with temperature, albeit weakly, it remains to assume that the establishment of a stationary signal is determined not only by the spin-spin interactions, but in part also by spin-lattice interactions. If the passage through resonance occurs within a time \( \Delta t \gg \tau_s \), then the form of the spectrum does not depend on the magnetic field sweep direction.

When the magnetic field is scanned continuously, the signal should depend (by virtue of the interaction between the packets) not only on the magnetization of the spin packets within the modulation interval \( H_2 \pm H_M \) but also beyond its limits. For this reason, the form of the spectrum varies with the direction of the field scanning, see Fig. 1. This means that the time variation of the signal after the field scanning is turned off also entails a change in the magnetization of the spin packets in a certain vicinity of \( H_2 \) somewhat larger than the modulation zone \( H_2 \pm H_M \). In the case of continuous scanning of the magnetic field, three principal processes are present: 1) loss of the magnetization of the spin packets within the interval \( H_2 \pm H_M \), 2) partial (or total) restoration of the spin packets that leave continuously (or have left) the zone \( H_2 \pm H_M \), 3) capture by the modulation zone (during the course of the spin scanning) of new packets that have at the initial period of time a maximum magnetization, and consequently should make

a somewhat larger contribution to the EPR signal (an analysis of the effects of passage through resonance for inhomogeneously broadened lines is presented in a paper by Feher\(^2\)). When the field scanning is turned off, the capture of new packets ceases, so that it can be assumed that the initial decrease of the signal, and its subsequent growth (see Fig. 2) are due to a superposition of processes of the type 1) and 2) (see above). Since there is presently no general theory of the effects of passage through resonance under AFP conditions in the presence of interaction between the packets (and, furthermore, we do not know the concrete mechanism of these interactions in the case of D centers), a quantitative description of the observed phenomena is impossible.

The foregoing qualitative explanation of the transient processes following the turning off of the field scanning agrees with the behavior of the signal \( \chi' \) following a jumpwise change of \( H_M \) (Fig. 4). The field scanning was turned off at the point A, with \( H_M = 0.03 \text{ Oe} \); after the stationary signal was established, \( H_M \) was increased jumpwise at the point B to 0.1 Oe. We see that the signal increases rapidly at first (within a time <1 sec), and then decreases smoothly with a time constant \( \tau^* \) to a stationary value. At the point C we have a jump of \( H_M \) from 0.1 to 0.05 Oe. The signal then decreases abruptly at first, and then increases smoothly to a stationary value.

When \( H_M \) is reversed, the width of the modulation zone \( H_2 \pm H_M \) changes jumpwise, and this leads to an abrupt change of the signal during the switching instant. The decrease of the signal with time after the switching of \( H_M \) (0.03 Oe to 0.1 Oe) is due, according to our assumption, to the loss of the magnetization of the "fresh" spin packets captured by the modulation zone \( H_2 \pm 0.1 \text{ Oe} \), and the increase of the signal after the switching of \( H_M \) (0.1 to 0.05 Oe) is due to partial restoration of the magnetization of the packets in the same interval \( H_2 \pm 0.1 \text{ Oe} \). We note that at all values of \( H_M \) the AFP conditions are satisfied quite rigorously, and since \( \tau^* \ll \tau_1 \), the loss of magnetization is due almost entirely to the interactions between the spin packets, and not to spin-lattice relaxation processes.

The anomalous behavior of the signals \( \chi' \) for D centers under AFP conditions (see Figs. 2 and 3) is due to the specifics of the structure of the EPR spectra of these centers. In fact, to explain the oscillations on the \( A_s = F(H_M) \) curves it is natural to assume that the width of the spin packets is \( \Delta H_1 \sim 0.2-0.3 \text{ Oe} \), and the time of passage through resonance \( \Delta t \sim \Delta H_1/H_M \omega_M \) decreases with increasing \( H_M \) for the considered packet, and consequently the amplitude of the registered signal harmonic also increases. Thus, the appearance of the first maximum on the \( A_s = F(H_M) \) curves corresponds to the condition when \( H_M \approx \Delta H_1 \sim 0.2-0.3 \text{ Oe} \). At \( H_M \sim \Delta H \) there appears a second maximum, since the modulation zone begins to capture the neighboring packets, etc. If \( H_M \approx \Delta H_1 \) or \( \Delta H \), then the spin-packet distribution can be regarded as quasiconstant, and the oscillations on the \( A_s = F(H_M) \) curves vanish accordingly. This is confirmed by the fact that in the case of inhomogeneously broadened lines due to the pointlike paramagnetic centers that are randomly distributed in the crystal (e.g., phosphorus atoms in silicon) or F

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centers in alkali-halide crystals), no such oscillations are observed in fact. For these centers, the distribution of the packets can be regarded as quasicontinuous for all the values of $H_M$ employed in practice\cite{5,6}. If we introduce the time of the spin-spin relaxation that determines the width of the spin packets, defined as $\tau_2 = 2\pi/\Delta H_1$, then we obtain $\tau_2 \sim 10^{-8}$ sec for the D centers.

In concluding this section, we note that when the spectrum was obtained with continuous field scanning it was impossible to resolve the hyperfine structure even for $H_M < 0.2$ Oe. The oscillations on the $A_1 = F(H_M)$ curves are also hardly noticeable (see Fig. 3). The apparent reason is that under continuous field scanning the interactions between the spin packets affect more effectively the value of the signal and lead to a "smearing" of the oscillations.

2. Temperature Measurements of the D-Center EPR Signal

Recognizing that in the one-dimensional magnetic system produced by the dislocations in silicon one cannot exclude the possibility of a phase transition\cite{3,4}, we have measured the temperature dependence of the D-center EPR signal. The measurements were made in the temperature interval 150–20° K. At lower temperatures, the slow-passage conditions were violated, and at higher temperatures the EPR signal was too weak.

It was assumed that the spectrum of the D centers consists of spin packets, each of which has a Lorentz shape. In this case $\chi'(H_0)$ takes the form (the summation is over the spin packets)

$$\chi'(H_0) = \sum_{i} \chi_{0i} \frac{1}{\pi H_{0i}} \sum_{H_0} g(H_0 - H_i) D(H_i - H_0),$$

(1)

where

$$D(H_i - H_0) = \frac{1}{\pi} \frac{1}{\pi H_{0i}} \sum_{H_0} g(H_0 - H_i) D(H_i - H_0),$$

(2)

$g(H_0 - H_i)$ is the spectral shape function, defined as $\chi_{0i} / \chi_0 = g(H_0 - H_i)$, $H_i$ is the resonance field of the $i$-th packet, $H_0$ is the average resonance field, $\chi_0$ is the static susceptibility averaged over the spectrum of one spin packet, $\chi_{0i}$ is the true static susceptibility of the spin packet, and $\gamma_i$ is the gyromagnetic factor.

For the integral intensity we have accordingly

$$I(H_0) = \int \chi'(H_0) dH_0 = \frac{1}{2} \sum_{i} \chi_{0i} \frac{1}{\pi H_{0i}} \sum_{H_0} g(H_0 - H_i) \frac{1}{1 + H_{0i}^2},$$

(3)

where $\tau_1 \approx \frac{2}{\gamma_i} T_{20} = \frac{1}{H_{0i}^2}$. Generally speaking, $\tau_1 \tau_2$ can be different for different spin packets. If we define the mean value of the parameter $\chi_{0i}$ by

$$\bar{\chi_{0i}} = \left[ \frac{1}{\gamma_i} \sum_{H_0} g(H_0 - H_i) \right]^{-1} \sum_{H_0} \chi_{0i} (H_0 - H_i)^{-1},$$

(4)

then we obtain for $I(H_0)$

$$I(H_0) = \frac{1}{2} \sum_{i} \chi_{0i} \frac{1}{\pi H_{0i}} \sum_{H_0} g(H_0 - H_i) \frac{1}{1 + H_{0i}^2},$$

(5)

where $\chi_{0i} = \sum \chi_{0i}$ is the magnetic static susceptibility of the entire spin system.

Writing down the spectrum at two values of $H_i$, namely $H_{i0}$ and $H_{i1}$, and measuring the saturation parameter $\rho = I(H_{i0}) / I(H_{i1})$, we can determine $\tau_1 \tau_2$:

$$\tau_1 \tau_2 = \frac{\rho - 1}{\rho \bar{H}_{0i}^2}.$$

(6)

We measured two quantities, $I(H_1 = 0)$ and $\rho$, after which we plotted $I(H_1 = 0) = F(1/T)$ and $\tau_1 \tau_2 = F(1/T)$. As seen from (5) $I(H_1 = 0) \sim \chi_{0i} \sim 1/ T$ if the spin system satisfies the Curie law. Since the amplitude of the experimentally observed signal is $A = \frac{\partial \chi}{\partial H_1} H_1$ then

$$I(H_1) = \frac{1}{H_1} \sum_{i} \gamma_i \chi_{0i} H_i.$$  

(7)

The double integration of the EPR signals as well as the entire subsequent reduction of the results were carried out with a computer. The maximum error in the determination of the relative values of $I(H_1)$ and $\tau_1 \tau_2$ did not exceed $\pm 10\%$ and is mainly connected with the signal/noise ratio in the registration of the EPR spectra.

To check on the sensitivity of the setup, we plotted simultaneously with the D-center signal the signal from CuSO$_4$·5H$_2$O; the latter satisfied the Curie law down to 20°K inclusive.

Figure 5 shows plots of $I(H_1 = 0)$ against the temperature for D centers at two sample orientations, $H_0 \parallel [111]$ and $H_0 \parallel [112]$. At temperatures higher than 45–50°K the experimental points agree satisfactorily with the Curie law: $I(H_0 = 0) \sim 1/T$. At temperatures 40–50°K there is an abrupt jump on the $I(0) = F(1/T)$ curve, and at $T < 50°$K an appreciable deviation from the Curie law is also observed. This behavior of the EPR signal gives grounds for assuming that a certain phase transition takes place in the dislocation spin system in the temperature interval 40–50°K. No significant change in the form of the spectrum is observed in the anomaly region. This is obviously due to the fact that the spectrum is inhomogeneously broadened. In fact, a change in the width of the spin packets $\Delta H_1$ exerts no significant influence on the width $\Delta H_C$ of the observed lines if $\Delta H_1 \ll \Delta H_C$.

Figure 6 shows the experimental plots of $\tau_1 \tau_2$ for D centers vs. 1/T. It is appropriate to note here that different parts of the spectrum are saturated somewhat differently with increasing microwave power. This means that for individual spin packets the value of $\tau_1 \tau_2$ can differ somewhat from the measured mean values of $\tau_1 \tau_2$. As seen from Fig. 6, in the 40–50° region (as is also the case for $I(H_1 = 0) = F(1/T)$ on Fig. 5) there is a singularity in the function $\tau_1 \tau_2 = F(1/T)$. It can be assumed that these singularities are connected with appearance of magnetic ordering (ferromagnetic or antiferromagnetic) in the dislocation spin system. The fact that $\tau_1 \tau_2$ is several times larger for
Ho $\parallel$ [111] than for Ho $\parallel$ [112] is obviously connected with the fact that at Ho $\parallel$ [112] the structure lines overlap much more strongly than at Ho $\parallel$ [111], and this leads to an increase of the cross relaxation, and hence to a decrease of $\tau_1 \tau_2$. Since measurements of $\tau_1$ in the temperature range 4.2—1.3°K have shown that it does not depend on the sample orientation, the difference in $\tau_1 \tau_2$ in Fig. 6 is probably due to the strong dependence of $\tau_2$ on the degree of overlap of the structure lines.

CONCLUSION

As a result of our investigations, we have established the following:

1. The D-center spectrum has a hyperfine structure. The presence of the hf structure leads to certain anomalies in the behavior of the signal $X'$ under AFP conditions.

2. The observed anomalies can be explained by assuming that an interaction having a characteristic time $\tau_3 \sim 3-10$ sec at temperatures 4.2—1.3°K exists between the hf structure lines or the spin packets of the spectrum. The presence of these interactions leads to a dependence of the shape of the spectrum on the direction in which the field Ho is scanned.

3. An anomalous temperature dependence was observed in the intensity of the D-center spectrum and in the behavior of the relaxation times $\tau_1 \tau_2$.

It is assumed that the strong deviation from the Curie law for the spectrum intensity, observed when the temperatures 40—50°K are approached, as well as the singularity in the dependence of $\tau_1 \tau_2$ on $1/T$, offer evidence in favor of the presence of a phase transition in the dislocation spin system.

To explain the character of the observed transition, we are continuing our measurements of the static magnetic susceptibility of the D-center system, and of the properties of the EPR spectra in the 8-mm band. It is obvious that once the character of the transition is explained, all the effects observed at temperatures below critical will be readily interpreted within the framework of a concrete spin model; in this case the hyperfine-structure lines can be connected with excitation of the spin waves by dislocation chains of spins that are in the ferro- or antiferromagnetic state, depending on the concrete character of this transition.

Translated by J. G. Adashko

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