Temperature dependence of conduction by reconstructed dislocations in silicon and nonlinear effects

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(Submitted 6 May 1988)
Zh. Eksp. Teor. Fiz. **95**, 183–191 (January 1989)

The intensities of combined-resonance lines from reconstructed dislocations in silicon were measured at temperatures from 1.3 to 40 K. A self-consistent analysis of the line intensity and line shape yielded the temperature dependence of the high-frequency mobility of the electrons along the dislocation. The effect of heating the system by a nonresonant microwave field on the amplitude and shape of the combined-resonance line was investigated. A change in the dielectric constant of a sample of deformed silicon crystal was observed at a frequency 9200 MHz on passing through conditions of combined-resonance at another frequency, and was investigated in the frequency range 8–19 GHz. This effect was used to observe at temperatures 1.4–4.2 K the generation of high harmonics (frequency doubling and tripling) with relative intensity 10^{-6}–10^{-8}.

A resonant change of the dielectric constant $\varepsilon = \varepsilon' + \varepsilon''$ of plastically deformed silicon single crystals was observed[1,2] when the condition $\hbar\omega = g\mu_B H_0$ was met [ω is the electric-field frequency at which $\varepsilon(\omega)$ is measured, H_0 is the static magnetic field, g is the spectroscopic splitting factor, and μ_B is the Bohr magneton]. It was shown that this singularity (called the *Ch* line[1,2]) is due to resonance transitions between the Zeeman levels of electrons trapped in a deep electronic state $E_{Ch} = E_C - 0.35$ eV produced by one type of dislocation. These resonance transitions are due to a term of the form $\alpha[\mathbf{P}\times\mathbf{S}]$ in the Hamiltonian, where $\mathbf{P}$ is the electron momentum along the dislocation. This term appears in the Hamiltonian as a result of spin-orbit interaction, since the dislocation core has no inversion center. Spin transitions brought about by terms of this type were named in Ref. 3 combined resonance, and the *Ch* line is the first observation of combined resonance in a one-dimensional system.

We carried out precise *Ch*-line measurements in a temperature range from 1.3 K to 40 K, and determined, by a self-consistent analysis of the *Ch*-line intensity and shape, the temperature dependence of the high-frequency mobility $\mu(\omega)$ of the electrons in the E_{Ch} state along the dislocations. In addition, we observed an influence of heating the system by a nonresonant microwave field on the amplitude and profile of the combined-resonance line, and also harmonic generation by interaction of a microwave field with dislocations.

SAMPLES AND EXPERIMENTAL TECHNIQUE

We investigated *n*-silicon samples doped with phosphorus at a density $2\cdot 10^{14}$ cm^{-3} and plastically deformed by 1.5% by compression along [110] at 700 °C; this corresponded to an integrated dislocation density $N_D \approx 10^9$ cm^{-2}. The samples were annealed for 30 min at $t = 850$ °C to eliminate EPR-active broken bonds in the dislocation cores.[4–6]

The dielectric constant $\varepsilon_\mu = \varepsilon'_\mu + i\varepsilon''_\mu$ of the sample was measured at 9200 MHz by placing it at the maximum of the electric field E_1 of a microwave cavity. The sample and cavity temperatures were equal, and the sample was painstakingly shielded against nonequilibrium optical radiation. The use of a heterodyne microwave oscillator and a superheterodyne microwave receiver permitted ε_μ to be measured at a microwave power of order 10^{-12} W entering the cavity.

A static magnetic field $\mathbf{H}_0 \| [001]$ was applied to the sample. To increase the sensitivity we used modulation of H_0 at a frequency 80 Hz and an amplitude ~0.1 Oe, and lock-in detection, so that the spectra of $\partial\varepsilon'_\mu/\partial H_0$ and $\partial\varepsilon''_\mu/\partial H_0$ could be recorded. The sample orientation corresponded to the maximum value of the combined resonance (of the *Ch* line), i.e., the microwave field $\mathbf{E}_1$ was parallel to the dislocations: $E_1 \| [1\bar{1}0]$ (see Refs. 1 and 2). To investigate the influence of a nonresonant microwave field on the magnitude and shape of the combined-resonance line, to investigate nonlinear effects, and also to observe effects due to overheating of the electron system, we applied to the system, in addition to the field $\mathbf{E}_1$, an additional microwave field $\mathbf{E}_2$ of frequency ω from another microwave oscillator. We used for this purpose either a coaxial lead-in terminated by a post placed in the cavity alongside the sample, or a two-mode microwave cavity which made it possible to attain a field intensity $E_2 \gtrsim 300$ V/cm at the second frequency. The sample was mounted in the cavity in such a way that the $[1\bar{1}0]$ direction made equal 45° angles with the directions of the fields E_1 and E_2.

TEMPERATURE DEPENDENCE OF THE *Ch* LINE AND INFLUENCE OF MICROWAVE PUMPING. EXPERIMENTAL RESULTS

In Ref. 2 we measured the temperature dependence of the combined-resonance intensity (of the *Ch* line) in the temperature range 10–30 K. The use of very low microwave

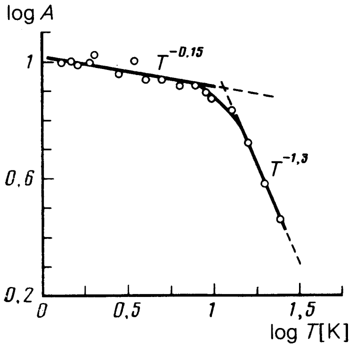

FIG. 1. Temperature dependence of the *Ch*-line intensity normalized to the EPR signal amplitude of the paramagnetic standard $CuSO_4\cdot 5H_2O$.

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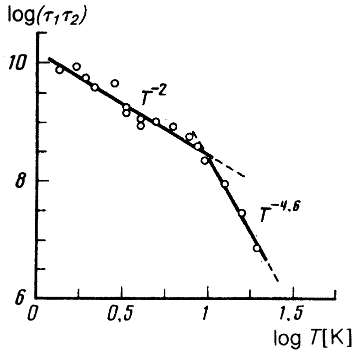

FIG. 2. Temperature dependence of the quantity $\tau_1\tau_2$ calculated from the dependences of the saturation of the combined-resonance line by a microwave field.

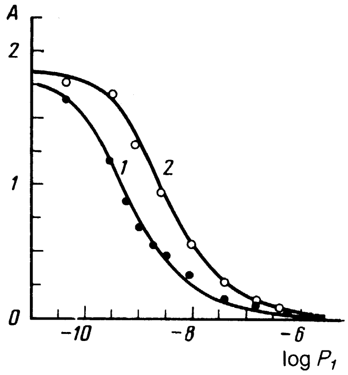

FIG. 4. Dependence of the *Ch*-line intensity on the microwave-field power P_1 in the absence (1) and presence (2) of microwave pumping at a nonresonant frequency; $T = 1.4$ K.

power in the present study has made possible measurements down to 1.4 K. Figure 1 shows the temperature dependence of the ratio of the intensities of the *Ch* line and of the EPR of a paramagnetic $CuSO_4 \cdot 5H_2O$ standard. At $T > 10$ K we have observed the $A_{Ch}/A_{Cu} \propto T^{-(1.3-1.4)}$ dependence cited in Ref. 2, which goes over smoothly into $A_{Ch}/A_{Cu} \propto T^{-0.15}$ for $T < 10$ K. Some difference between our present results and those of Ref. 2 is due to the difference between the samples.

The data of Fig. 1 correspond to $E_1 \to 0$. With increase of E_1, the intensity of the *Ch* line begins to decrease and its width increases.[2] Application of an equal electric field but with nonresonant frequency ω_2 does not change the *Ch*-line amplitude. This means that the discussed *Ch*-line saturation is not due to overheating of the electron system or of the system of quasilocal phonons, but is due to overheating of the spin system of the quasi-one-dimensional electrons. Effects of overheating of the electron system were observed only in much stronger electric fields and will be discussed below.

The dependence of A_{Ch} on E_1 during the initial saturation stage is well described by the expression $A_{Ch} = A^0_{Ch}(1 + \alpha E_1^2)^\beta$. The value of β at $T > 3$ K depends little on T and is equal to -0.5, while in the 3–1.4 K region it decreases from -0.5 to -1.2. The parameter α is proportional to $A_{Ch}\tau_1\tau_2$. Figure 2 shows the temperature dependence of $\alpha/A_{Ch} \propto \tau_1\tau_2$.

Figure 3a shows the experimental temperature dependence of the shapes of the resonance curves. Figure 4 shows the dependences of the *Ch*-line intensity on the "measurement" power P_1 at a frequency $\omega_1/2\pi = 9300$ MHz in the absence (1) and presence (2) of a microwave pump with $P_2 = 0.5$ W and frequency $\omega_2/2\pi = 17$ GHz, while Fig. 5 shows the change of the *Ch*-line shape under the influence of the microwave pump (at 1.4 K). For simplicity, these measurements were made after a brief illumination of the sample with white light, which decreased by approximately an order of magnitude the constant α that characterizes the *Ch*-line saturation. The *Ch*-line shape was also changed thereby.

DISCUSSION OF RESULTS

The results confirm the conclusions of Ref. 2 that the observed combined resonance is due to electrons trapped in a deep one-dimensional band connected with one of the types of reconstructed dislocations. To explain the temperature dependences of the shape and amplitude of the line it must be

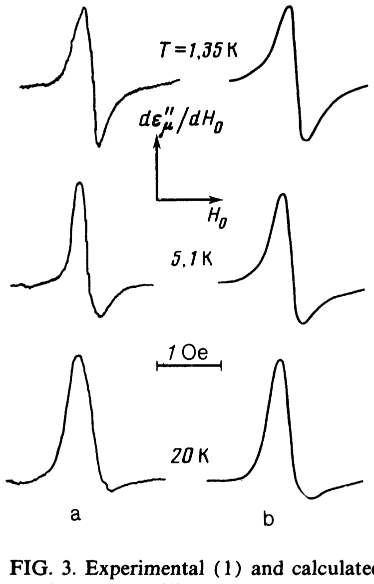

FIG. 3. Experimental (1) and calculated (b) shapes of $\partial\varepsilon''_\mu/\partial H_0$ resonance curves at different temperatures.

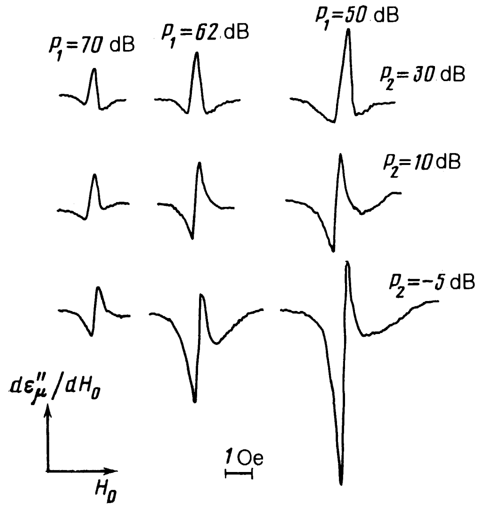

FIG. 5. Change of *Ch*-line shape by simultaneous action of resonance (P_1) and nonresonance (P_2) fields; $T = 1.4$ K.

assumed that the electrons can move along finite dislocation segments of length L and that their mobility $\mu(\omega)$ on these segments decreases with rise of temperature, i.e., is limited by scattering from phonons. Detailed calculations of these models are contained in Ref. 7 for $\sigma(\omega)=\omega\varepsilon''(\omega)/4\pi$ we have the expression

$$\frac{\sigma(\omega)}{\mathscr{L}_D}=eN\mu(\omega)\left\langle S\left(\frac{L}{L_D}\right)\right\rangle_L -i\frac{N}{8kT}\left\langle\frac{(\mu(\omega)S(L/L_D)mv)^2\Omega^2}{\omega-\Omega+i/\tau_2+iD(mv)^2S(L/L_s)/2(1-i\omega\tau)}\right\rangle_L, \quad (1)$$

where $S(x)=1-e^{i\pi/4}\tanh(xe^{-i\pi/4})/x$ is a structure factor, $\mathscr{L}_D$ is the total length of the conducting segments per unit volume, and $\langle\ldots\rangle_L$ denotes averaging over the lengths L of the conducting segments [we use an averaging with a Poisson weight

$$p(L)=\exp(-L/L_D)/L_0),$$

N is the electron density per unit dislocation length, $\mu(\omega)=e\tau/m(1-i\omega\tau)$ is the high-frequency mobility, $D=T\tau/m$ is the diffusion coefficient,

$$L_D=[(D+e\mu N\ln(L^2/b^2)/\varepsilon)/\omega]^{1/2},$$

where b is a quantity on the order of the atomic dimension, $L_S=(D/\omega)^{1/2}$, and $\Omega=g\mu_BH_0/\hbar$ is the resonance frequency.

To understand the mechanism of the dislocation conductivity of the electrons, it is important to obtain its temperature dependence. It follows from (1) that the square root of the combined-resonance amplitude is connected with the one-dimensional conductivity σ_d by the approximate relation

$$A^{1/2}=C\frac{\sigma_d}{\sigma_0}S_\sigma\left(\frac{\sigma_d}{\sigma_0}\right), \quad (2)$$

where $\sigma_d=e\mu N$, $S_\sigma=(\langle|S(L/L_D)|^2\rangle)^{1/2}$, C is a temperature-independent constant, and $\sigma_0=\omega L_0\varepsilon_0/\ln(L_0^2/b^2)$ is the characteristic conductivity at which depolarization effects become substantial. Using the characteristic values $L_0=500$–1000 Å, $\ln(L_0^2/b^2)=10$, and $\varepsilon_0=12$, we obtain $\sigma_0\approx10^{-12}$–10^{-11} cm/Ω at the experimental frequency $\omega/2\pi=9200$ GHz. We have assumed $D\ll\sigma_d\ln(L^2/b^2)/\varepsilon_0$, which is valid for low temperatures.

The relation (2) is plotted in Fig. 6. At low values of the conductivity ($\sigma_d\ll\sigma_0$) the square root of the amplitude is proportional to σ_d. With increase of the conductivity, the amplitude becomes saturated as a result of depolarization effects.

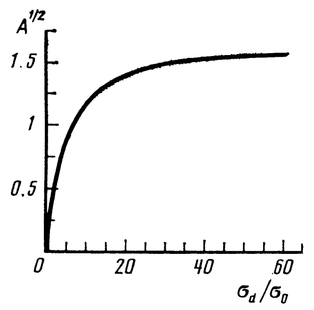

FIG. 6. Calculated dependence of the combined-resonance amplitude on the ratio σ_d/σ_0: σ_d is the one-dimensional conductivity and σ_0 is the characteristic conductivity at which depolarization effects become substantial.

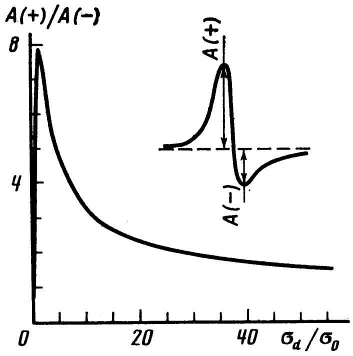

FIG. 7. Calculated characteristic $A(+)/A(-)$ of the combined-resonance line shape vs the ratio σ_d/σ_0.

The conductivity cannot be calculated directly from (2) in view of the presence of the undetermined coefficient C. To estimate this coefficient, and also for an independent estimate of the conductivity, we can use the combined-resonance line shape.

Owing to the complex structure factor $S(x)$, resonance absorption receives contributions from both the real and imaginary parts of the resonance magnetic susceptibility.[2,7] In this case the line shape depends only on the ratio of the real and imaginary parts of the structure factor, a ratio uniquely determined by σ_d/σ_0. Figure 3a shows the experimental shapes of the $\partial\varepsilon''_\mu/\partial H_0$ lines at temperatures $T=1.35$, 5.1, and 20 K, while Fig. 3b shows the calculated line shapes for the parameter values $\sigma_d/\sigma_0=100$, 25, and 6.25, respectively.

The simplest characteristic of the line shape is the ratio of the resonance-absorption maximum amplitude $A(+)$ to the minimum $A(-)$. Figure 7 shows the calculated dependence of this ratio on σ_d/σ_0. This curve and relation (2) make it possible to estimate the temperature dependence of the conductivity σ_d by two methods, with the coefficient C

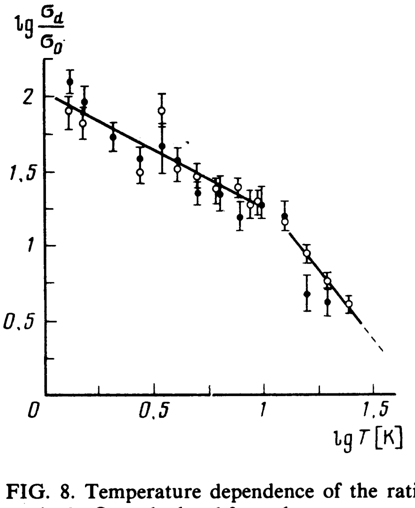

FIG. 8. Temperature dependence of the ratio σ_d/σ_0 obtained by two methods: ●—calculated from the resonance-curve shape; ○—calculated using Eq. (2).

in (2) chosen such that the calculated line shapes were close to the experimental for all temperatures (see Figs. 3a and 3b). The dependences obtained in this manner are shown in Fig. 8. It can be seen that the power-law dependence $\sigma_d/\sigma_0 \propto T^{-1.5}$ ($T > 10$ K) goes over with decrease of temperature into a dependence of the type $\sigma_d/\sigma_0 \propto T^{-0.6}$.

The dependence of the *Ch*-line width on the microwave power[2] attests to a strong inhomogeneous broadening of the line. The line saturation is also influenced by the "entanglement" of the real and imaginary parts of the resonance susceptibility. In addition, the saturation is inhomogeneous, with short dislocation segments saturated at higher powers than long ones. The large number of factors influencing the saturation hinders greatly a quantitative reduction of the dependence of the *Ch*-line amplitude on the microwave field E_1. This makes, in general, the data obtained for $\tau_1\tau_2$ from the *Ch*-line saturation not very reliable, especially at low T, when the factor β depends on T. We shall nonetheless assess these data. It is reasonable to assume (as is confirmed by the data of Ref. 2) that $\tau_2 \approx \tau_1$ in the investigated system. It follows then from Fig. 2 that $\tau_1 \propto T^{-1}$ for $T < 10$ K, as is typical of single-phonon relaxation processes. For $T > 10$ K we have $\tau_1 \propto T^{-2.3}$. Nearly equal dependences were obtained in Refs. 8 and 9 for spin-lattice relaxation of dislocation broken bonds. This was attributed in Refs. 8 and 10 to the large contribution made to the spin-lattice relaxation by quasilocal vibrational modes of the dislocation core, something quite reasonable in this case, when we are dealing with electrons that are strongly localized in a direction perpendicular to the dislocation.

We discuss now the influence of high-intensity microwave pumping. As follows from Fig. 5, the *Ch*-line shape is noticeably changed in this case and assumes the form of an inverted absorption curve. It follows from Eq. (1) that this change of shape can take place if the ratio of $\mu(\omega)$ and L changes, namely if $\mu(\omega)$ decreases and/or L increases. This is the opposite of the change of $\mu(\omega)$ and L when the spin system is heated. The saturation curves $A_{Ch}(P_1)$ shift to the right (see Fig. 4), corresponding to an approximately fivefold decrease of the parameter $\alpha \approx \tau_1\tau_2[\mu m v S(L/L_D)]^2$. The results can be understood if it is assumed that the microwave field E_2 heats the electrons in a one-dimensional band and increases the localization length L. This is simultaneously accompanied by some heating of the quasilocal-phonon system, which leads to a decrease of $\mu(\omega)$ and τ_1. What is still not understood is the strong distortion of the shape of the *Ch* line if strong E_2 and E_1 are simultaneously present. The *Ch* line acquires in this case a broad "pedestal" (see Fig. 5).

CHANGE OF DISLOCATION CONDUCTIVITY ON SATURATION OF THE COMBINED RESONANCE

Figure 9 shows the observed change of $\varepsilon_\mu(\omega_1)$ on going through resonance conditions at a frequency ω_2, i.e., in a field $H_0 = \hbar\omega_2/g\mu_B$ (we name this line *Ch* *). A plot of the combined-resonance line (*Ch* line) observed in a field $H_0 = \hbar\omega_1/g\mu_B$ is shown on the right for comparison. The dependence of the *Ch* *-line amplitude on the pump field E_2 is well described by the expression

$$A_{Ch^*} = A^0_{Ch^*}\alpha E_2^2(1+\alpha E_2^2)^{-1}, \qquad (3)$$

where α is a coefficient that is constant at the given tempera-

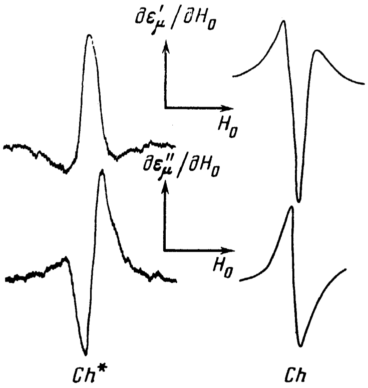

FIG. 9. Change of $\varepsilon_\mu(\omega_1)$ on passing through the combined-resonance conditions at a frequency ω_2: $\omega_1/2\pi = 9200$ MHz. Right—resonance curves (*Ch* line) for combined resonance at $\omega/2\pi = 9200$ MHz; $T = 1.4$ K.

ture. The *Ch* *-line intensity $A^0_{Ch^*}$ is $(3–5)\cdot 10^{-3}$ of the intensity A^0_{Ch} of the *Ch* line and depends weakly on ω_2 in the interval $\omega_2/2\pi = 8–19$ GHz (see Fig. 10). Figure 11 shows the temperature dependence of A_{Ch^*}.

Let us discuss the results. It follows from them that the *Ch* line corresponds to a resonant decrease of the electron mobility $\mu(\omega)$ in a one-dimensional band when $\hbar\omega$ is equal to the Zeeman splitting $g\mu_B H_0$ of the spin levels of these electrons. This decrease of $\mu(\omega)$ is due to the resonant energy transfer from the electron translational motion to its spin degree of freedom.

From the fact that the *Ch* *-line shape is similar to that of the *Ch* line, but inverted, and from the dependence of the *Ch* *-line amplitude on the "pump" power at the frequency ω_2; it follows that *Ch* * corresponds either to an increase of $\mu(\omega_2)$ or to a decrease of the localization length L on saturation of the combined resonance by the pump E_2. (Note that $\alpha/A_{Ch^*} \propto T^{-2}$ and coincides with $\tau_1\tau_2$ calculated using the *Ch*-line saturation.)

We discuss now the nature of the *Ch* * line. Saturation of the combined resonance, first, equalizes the electron spin-sublevel populations and, second, can overheat the quasilocal vibrational modes via spin-lattice relaxation. Recognizing that a dependence $\mu(\omega) \propto T^{-1.4}$ is observed in experiment, it is unlikely that overheating of the quasilocal phonons can cause an increase of $\mu(\omega_1)$. It is also unlikely (albeit not excluded) that overheating of the quasilocal phonons will decrease the localization length L.

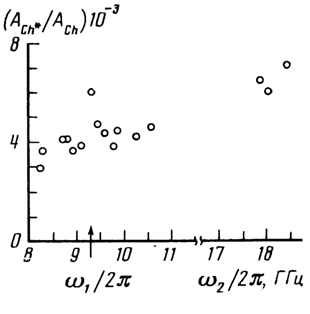

FIG. 10. Dependence of the amplitude ratio A_{Ch^*}/A_{Ch} on the frequency ω_0; $T = 1.4$ (A_{Ch} for 9200 MHz).

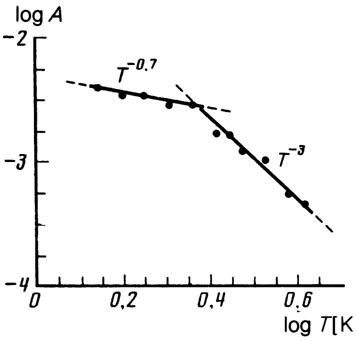

FIG. 11. Temperature dependence of the *Ch* *-line amplitude.

One possible explanation of the *Ch* * line is the following: owing to the absence of an inversion center and of spin–orbit interaction in this system, a connection exists between the electron motion along a dislocation and its spin degree of freedom, therefore processes take place which lead at positive spin temperature to deceleration of the electrons and to a decrease of $\mu(\omega_1)$. When the resonance saturates, the spin contribution to $\mu(\omega_1)$ vanishes and this should lead to an increase of $\mu(\omega_1)$.

In the simple model of Ref. 7, however, this effect should decrease rapidly with increase of $\Delta\omega = |\omega_1 - g\mu_B H_0/\hbar|$ and become much smaller than the experimental value of the *Ch* * line even for $\Delta\omega \geqslant 0.3$ GHz. This means probably that there exists an additional mechanism, not accounted for in the model of Ref. 7, which contributes to the effect and depends weakly on $\Delta\omega$.

We are thus still unable to explain unambiguously the high intensity of the *Ch* * line at $\Delta\omega \geqslant 0.3$ GHz.

GENERATION OF MICROWAVE HARMONICS

If the dislocation conductivity is nonlinear, application of a field E of frequency ω should produce not only an electron-momentum component of frequency ω, but also components of frequency 2ω, 3ω, etc:

$$\langle v\rangle = \mu_1(\omega)E_0\exp(i\omega t) + \mu_2(\omega)E_0^2\exp(2i\omega t) + \dots$$

One of the causes of the nonlinearity is the absence of an inversion center in the system, which leads to combined resonance. This should cause appearance of a resonance response $\partial\varepsilon_\mu(\omega)/\partial H_0$ in fields H_0 corresponding to the frequencies 2ω, 3ω,.... Observations of the *Ch* * line makes it easy to reveal the onset of both odd and even harmonics of the microwave field applied to the sample by recording the singularities of $\partial\varepsilon_\mu/\partial H_0$ in fields H_0 corresponding to combined resonance at the frequencies 2ω, 3ω, etc. We have actually observed such singularities at helium temperatures.

That frequency doubling and tripling takes place only in the sample can be proved by several facts. First, the amplitudes of the $Ch^*_{2\omega_1}$ and $Ch^*_{3\omega_1}$ lines are not changed by placing in the microwave channel filters that attenuate the higher harmonics by 60 dB. Second, simultaneous application of frequencies ω_1 and ω_2 to the sample resulted in a resonance singularity in a magnetic field $H_0 = \hbar(\omega_1 + \omega_2)/g\mu_B$. Generation of the frequency $(\omega_1 + \omega_2)$ somehow outside the sample is practically excluded in this case by the experimental setup. Third, the proved premise follows from an analysis of the anisotropy, measured by us, of the $Ch^*_{2\omega_1}$ amplitude.

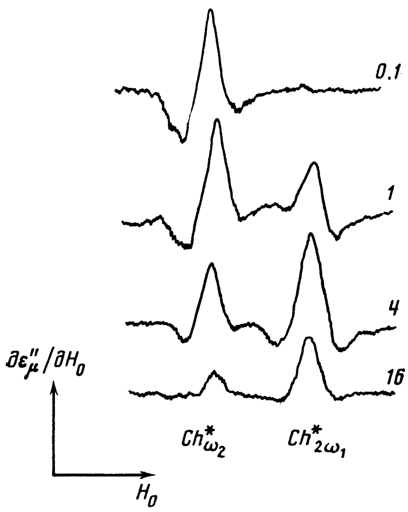

FIG. 12. Experimental plot of the change of $\varepsilon_\mu(\omega_1)$ on going through the resonance conditions for the frequencies ω_2 and $2\omega_1$ at different intensities of the "measuring" field E_1 (the right-hand side numbers in the figure).

Figure 12 shows by way of illustration plots of the *Ch* * line corresponding to saturation of combined resonance of the second harmonic of the "measuring" microwave field $\omega_1/2\pi = 9330$ MHz. A pump of frequency $\omega_2/2\pi = 18\,653$ MHz was simultaneously applied to the sample and made possible observation of the $Ch^*_{\omega_2}$ line. It can be seen that increasing the power of frequency ω_1 produces a line corresponding to the second harmonic of ω_1. The observed decrease of the $Ch^*_{\omega_2}$ line corresponding to heating of the electron system by the field of frequency ω_1 which decreases the degree of saturation of the spin system by the pump field ω_2 (see Ref. 3).

Comparing the dependences of intensities of the lines $Ch^*_{\omega_2}$, $Ch^*_{2\omega_1}$, $Ch^*_{3\omega_1}$ etc. on the pump power, we can roughly estimate the harmonic-generation efficiency. At $T = 1.4$ K, the *Ch* * line corresponding to the pump frequency ω_2 reaches a maximum amplitude in a pump field of order $E_1 = (3\text{–}5)\cdot 10^{-3}$ V/cm. Approximately the same second- and third-harmonic amplitudes are reached in a pump field $E_1 = (5\text{–}10)$ V/cm. Thus, the nonlinear terms in the dislocation conductivity are quite large. The harmonic-generation efficiency depends little on T in the interval 1.4–4.2 K.

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Translated by J. G. Adashko