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## Anisotropy of the scattering of conduction electrons by dislocations in aluminum

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The de Haas-van Alphen effect was used to measure the broadening  $\Gamma$ , due to electron scattering by dislocations, of the Landau levels of the electrons of the  $\beta$  and  $\gamma$  sections of the Fermi surface of aluminum. The dislocations were produced by uniaxial dilatation of the samples directly in the course of the measurements at 1.3 K, at a load  $\sigma \parallel [111]$ , and in a magnetic field **B** \parallel [110]. It was observed that  $\Gamma$ depends strongly on the position of the electron orbit on the Fermi surface and on the dislocation density. A ratio  $\Gamma_{\gamma}/\Gamma_{\beta} = 3.5$  is obtained for dislocation densities  $D \sim 10^8$ , and decreases to 2 with increasing D. An increase in the areas S of the extremal sections of the Fermi surface is observed at the same time when the degree of uniaxial deformation  $\Delta V/V$  is increased. The values of  $\mu = -(3/2)(\Delta S/S)/(\Delta V/V)$ were measured and found to be 90 and 60 for the  $\beta$  and  $\gamma$  sections, respectively. These values of  $\mu$  were used to calculate the anisotropy of the broadening of the Landau levels in accord with Watts' theory [Phys. Cond. Matt. 19, 125 (1975)]. The values obtained are in satisfactory agreement with the results of the direct measurements.

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#### 1. INTRODUCTION

We have investigated the scattering of conduction electrons by dislocations in aluminum with the aid of a procedure based on the measurements of the amplitude of the de Haas-van Alphen effect, followed by calculation of the Dingle temperature<sup>[1]</sup> that characterizes the collision broadening  $\Gamma$  of the Landau levels. Particular attention was paid to the study of the relation between the Dingle temperature for electrons on different sections of the Fermi surface, a relation governed by the anisotropy of the scattering in momentum space.

In the case of electron scattering by point defects, the Dingle temperature  $\chi_i$  and the (non-transport) electron collision frequency  $\tau_i^{-1}$  in a zero magnetic field, averaged over the i-th electron orbit, are in essence equivalent characteristics of the scattering<sup>1</sup>:

$$X_i = \Gamma_i / \pi k = \hbar / 2\pi k \tau_i.$$
(1)

The connection between the Dingle temperature and the characteristics of electron scattering by dislocations in a zero magnetic field has not yet been rigorously established. We shall therefore regard the measured Dingle temperature as an autonomous characteristic of the scattering, and use relation (1) to calculate the effective frequency of the collisions with the dislocations in a magnetic field when we compare the obtained data with the results of the investigation of scattering by other methods.

The dependence of the Dingle temperature on the

electronic characteristics and on the parameters of the dislocation system was investigated theoretically in a number of papers.<sup>[4-10]</sup> This dependence is clearly seen from the Watts formula<sup>[7]</sup> calculated on the basis of the mechanism of "dephasing of the quantum oscillations":

$$X_{i} = \frac{\hbar e F_{i}^{2}}{k c m_{e}^{i} B} \begin{cases} \langle \mu \rangle_{i}^{2} \overline{\varepsilon^{2}}, & \xi_{i} = R_{i}^{2} D \ll 1, \\ \langle \mu^{2} \rangle_{i} \overline{\varepsilon^{2}} l / L_{i}, & \xi_{i} \gg 1, \end{cases}$$
(2)

where B is the magnetic field, D is the dislocation density, R is the Larmor radius, F is the oscillation frequency,  $\epsilon = \Delta V/V$  is the change of the volume in the uniaxial deformation, l is the correlation length (see<sup>[7]</sup>). L is the length of the classical electron trajectory, and  $\Delta F/F = -(2/3)\mu\epsilon.$ 

If we assume that  $\overline{\epsilon^2} \sim D$  and  $l \sim D^{-1/2}$  then, as seen from (2),  $X \sim D^{1/2}$  in the case of large dislocation densities  $(\xi \gg 1)$  and  $X \sim D/B$  in the case of small dislocation densities ( $\xi \ll 1$ ). This agrees with the results of Vinokur,<sup>[8]</sup> who calculated the probability of the scattering of conduction electrons by dislocations in the Born approximation.

It should be noted that the quantities  $\overline{\epsilon^2}$  and l in formulas (2) are not uniquely determined by the dislocation density D. Thus, for example, the formation of dislocation dipoles decreases  $\overline{\epsilon^2}$ , although the dislocation density may not change in this case. For this reason, and also because the existing experimental methods of determining the dislocation density are not accurate enough, the comparison of the experimental and theoretical dependences of the Dingle temperature on the dislocation density is apparently not a sufficiently reliable means of verifying the theory.

However, as seen from formulas (2), the anisotropy of the scattering of the conduction electrons by dislocations, i.e., the ratio of the Dingle temperatures of different electron groups on the Fermi surface in the limiting cases of high and low dislocation densities, depends much less on the parameters of the dislocation system and is determined mainly by the electronic characteristics of the metal:

$$\frac{X_{\alpha}}{X_{\beta}} \rightarrow \frac{\langle \mu \rangle_{\alpha}^{2} F_{\alpha}^{2}}{m_{e}^{\alpha}} \frac{m_{e}^{\beta}}{\langle \mu \rangle_{\beta}^{2} F_{\beta}^{2}}, \quad D \rightarrow 0,$$

$$\frac{X_{\alpha}}{X_{\beta}} \rightarrow \frac{\langle \mu_{\alpha}^{2} \rangle F_{\alpha}^{2}}{L_{\alpha} m_{e}^{\alpha}} \frac{L_{\beta} m_{e}^{\beta}}{\langle \mu_{\beta}^{2} \rangle F_{\beta}^{2}}, \quad D \rightarrow \infty.$$
(3)

If a set of samples with different dislocation densities is available, it is possible to trace the variation of the scattering anisotropy and make a quantitative comparison with the theory, after first determining all the electronic parameters that enter in (3). The purpose of the present work was in fact to carry out such a measurement program.

In parallel with the measurements of the Dingle temperatures, we determined also the transport frequency  $\tau_H^{-1}$  of the electron collisions from the helicon damping.<sup>[11]</sup> Comparison of the absolute values of  $\tau_H^{-1}$  and  $\tau_l^{-1}$  leads to conclusions on the dependence of the electron-scattering probability on the scattering angle.<sup>[12, 13]</sup>

#### 2. PROCEDURE AND CONDITIONS OF EXPERIMENT

1. Determination of the Dingle temperature. The de Haas-van Alphen effect was registered by the "helicon generator" procedure first proposed by us for this purpose.<sup>[14]</sup> The sample in the form of a plane-parallel plate was placed in crossed coils. The primary and secondary coils were connected respectively to the output and input of an amplifier having a sufficiently high gain and provided with a phase shifter. When positive feedback is produced, such a system generates oscillations at the frequency of the first helicon resonance<sup>[15]</sup> that corresponds to excitation of helicon of fixed wavelength  $\lambda = 2d$  in the sample (d is the sample thickness).

In the absence of quantum effects, the generation frequency varies linearly with the magnetic field. In the quantum region, quantum oscillations are superimposed on the linear frequency dependence. In our geometry, the relative value of the frequency oscillations is according to<sup>[6]</sup>

$$\frac{\Delta f}{f} = 2\pi \frac{\partial M}{\partial B} \sin^2 \theta, \tag{4}$$

where  $\theta$  is the angle between external magnetic field B and the normal to the sample surface.

An important fact from the experimental point of view is that relation (4), which connects the oscillating magnetic susceptibility  $\partial M/\partial B$  with the generation frequency, does not enter in the instrumental factors. This affords a unique opportunity of measuring the absolute value of the amplitude  $\partial M/\partial B$  of the oscillations in the de Haas-van Alphen effect without additional calibrations. The oscillating quantity is the frequency, which is readily measured with high accuracy.

According to the Lifshitz-Kosevich theory,<sup>[17]</sup> when account is taken of scattering,

$$\frac{\partial M}{\partial B} = A \frac{T \exp\left(-2\pi^2 k X/\hbar\omega_c\right)}{B^{3/2} \operatorname{sh}\left(2\pi^2 k T/\hbar\omega_c\right)} \cos\frac{2\pi F}{B},\tag{5}$$

where

$$A = \left(\frac{2e}{\hbar c}\right)^{\frac{s_{1}}{h}} \pi^{\frac{1}{h}} kF^{2} \cos\left(\frac{\pi g_{eff} m_{e}}{2m_{o}}\right) \left|\frac{\partial^{2}S}{\partial k_{z}^{2}}\right|^{-\frac{1}{h}},$$
$$\omega_{c} = eB/m_{c}c.$$

The Dingle temperature X, as can be easily seen from (4) and (5), can be determined from the slope of the plot of

$$\ln G = \ln \left[ \frac{\Delta f}{f} \operatorname{sh} \left( \frac{2\pi^2 kT}{\hbar \omega_c} \right) B^{t/2} \right]$$

against the reciprocal field 1/B. Typical plots of lnG for two types of oscillations in aluminum are shown in Figs. 1a and 1b.

Knowing simultaneously the absolute value of  $\partial M/\partial B$ , the Dingle temperature, and the cyclotron mass we can calculate the coefficient A in formula (5); this coefficient is a constant for a given trajectory in a given host metal and does not depend on the prior history of the sample. It turned out that  $A_{\gamma} = 1.6 \cdot 10^9 (m_c^{\gamma} = 0.13 m_0^{[18]})$ ,  $A_{\beta} = 1.7 \cdot 10^8 (m_c^{\beta} = 0.118 m_0^{[18]})$ . Knowledge of  $A_{\gamma}$  and  $A_{\beta}$ makes it possible to calculate the Dingle temperature in any sample if the temperature T and the absolute amplitude  $\partial M/\partial B$  are known. This was used by us to determine the increment of the Dingle temperature when the sample was deformed.

2. Determination of the transport frequency of the collisions. The transport frequency was determined from the Q of the helicon resonance using the formula

$$\tau_{II}^{-1} = eB/2Qm_cc.$$

The cyclotron mass is  $m_c = 9.1 \times 10^{-28}$ g. Particular attention was paid to the decrease of the contribution made to the damping by nonlocal mechanisms, which are capable of increasing substantially the error in the



FIG. 1. Dingle plots. Dependence of  $\ln G$  (see the text) on the reciprocal field for two types of oscillations in aluminum: a)  $\gamma$  oscillations in the third zone of the Fermi surface, b)  $\beta$  oscillations.



### measurements of $\tau_{H}^{-1}$ .<sup>[19]</sup>

3. Samples, deformation, determination of  $\mu$ , magnetic field. The oriented single-crystal samples were grown in a dismountable graphite mold, from aluminum with a resistivity ratio  $\rho(300K)/\rho(4.2K) = 20\ 000$ .

The samples were strips measuring  $0.8 \times 4 \times 40$  mm. The sample orientation is shown in Fig. 2. To prevent the metal from sticking to the mold, CO<sub>2</sub> gas ( $p \approx 50$ Torr) was admitted into the vacuum chamber prior to filling the mold with the metal.<sup>[20]</sup> When the sample was cooled in the mold, the difference between the thermal expansion coefficients of the aluminum and graphite produced stresses in the metal; stresses were produced also in the course of removal of the sample from the mold. To relieve these stresses, the finished samples were annealed in vacuum for 3–5 hours at 500°C.

No stacking faults or large-angle grain boundaries were observed in the samples prepared in this manner, and the dislocation density was  $\sim 10^8$  cm<sup>-2</sup>. A typical electron-microscope photograph of an undeformed sample is shown in Fig. 3.

The Dingle temperatures of  $\beta$  and  $\gamma$  electrons in the third zone of the Fermi surface of aluminum<sup>[21]</sup> were measured directly in the course of unaxial deformation of the samples at 1.3K. As shown earlier,<sup>[22]</sup> the Dingle temperature under these conditions is determined by the electron scattering by the dislocations, whose density increases with increasing degree of the plastic de-



FIG. 3. Typical electron-microscope photograph of the original sample. Individual dislocation lines can be seen, as well as circular orbits of electrons having areas corresponding to the  $\beta$  and  $\gamma$  electrons in fields of 8000 G (1), 10000 G (2) and 15000 G (3).

formation. Thus, in our experiments the measurements were made in fact on a large set of samples with different dislocation densities, starting with the usual  $D \sim 10^7 - 10^8$  cm<sup>-2</sup> and ending with relatively high densities at which the de Haas-van Alphen effect is not longer observed.

The important fact was that only the defect structure of the sample was altered in the course of the experiment, but the geometry of all the electric and magnetic fields and their orientations relative to the crystallographic axes and the sample axes remained unchanged. This increased significantly the measurement accuracy, especially in the investigation of the behavior of the anisotropy of the Dingle temperature with increasing dislocation density.

The samples were deformed under creep conditions. A tension force ranging from 0 to 3 kgf/mm<sup>2</sup> was applied to the sample in steps of approximately 0.05-0.1 kgf/mm<sup>2</sup>. During each step, the load was fixed and the electronic characteristics of the samples were measured. The deformation geometry is shown in Fig. 2. The deforming unit made it possible to deform the sample directly in the liquid-helium cryostat at decreased pressure. Great care was taken in the design of the deforming unit to keep the sample from rotating about the tension axis during the deformation.

Besides the irreversible decrease of the amplitude of the quantum oscillations in the course of deformation, which corresponded to an increase of the electron Dingle temperature, we observed a reversible change of the phase of the oscillations (Fig. 4), due to the change of the areas of the extremal sections of the Fermi surface. This has made it possible to calculate, by the procedure described, e.g., in <sup>[23]</sup>, the parameters  $\mu$  that characterize the sensitivity of the investigated cross sections to the uniaxial deformation and are close in character to the parameters  $\langle \mu \rangle$  that enter in formulas (2) and (3). A magnetic field up to 20 kG was produced



FIG. 4. Typical experimental plots of the oscillation frequency of a helicon generator at various loads. The changes of the amplitudes and phase of the oscillations can be seen. Short period— $\gamma$  oscillations, long period— $\beta$  oscillations.

with an electromagnet. The field homogeneity was not worse than  $10^{-5}$  over the sample dimensions. The current-control system made it possible to build up the field at a rate 1-50G/sec.

# 3. EXPERIMENTAL RESULTS AND THEIR DISCUSSION

1. Characteristics of electron scattering in the initial samples. The electron Dingle temperatures depended strongly on the sample preparation procedure, and no correlation whatever was observed between these temperatures and the purity of the metal, the latter being determined from the resistivity ratio  $\rho(300K)/\rho(4.2K)$ . The transport collision frequency  $\tau_{H}^{-1}$ , measured with the aid of the helicons, was on the contrary strongly dependent on the purity of the metal (it decrease monotonically with increasing resistivity ratio of the sample) and depended little on the sample preparation conditions. The Dingle temperatures of the  $\beta$  and  $\gamma$  electrons in three initial samples prepared by the indicated method were respectively  $0.2 \pm 0.1$ K and  $0.7 \pm 0.1$ K. The transport frequency  $\tau_{H}^{-1}$  in these samples was  $6 \times 10^{9}$  sec<sup>-1</sup>, corresponding to X = 0.007K.

If the sample is subjected to an annealing similar to that described in Nøst's method,<sup>[24]</sup> wherein the dislocation density is decreased, then the oscillation amplitude increases sharply (the Dingle temperature of the  $\gamma$  electrons is decreased to approximately one-fourth), but the helicon damping remains practically unchanged. The Dingle temperature of the  $\gamma$  electrons of samples that were deformed and then annealed by the Nøst method<sup>[24]</sup> was  $0.2 \pm 0.1$ K, and  $\tau_H^{-1} \approx 6 \times 10^9$  sec<sup>-1</sup>.

It can thus be stated that the transport collision frequency in our initial samples is determined mainly by scattering from impurities, while the Dingle temperature is determined by scattering from dislocations and their clusters.

This conclusion agrees with the notion that the dependence of the scattering probability W on the scattering angle  $\theta$  in collisions of electrons with dislocations has a sharp small-angle peak. If it is assumed that electron scattering through an angle  $\theta_0 \sim 1/N$  (N is the number of Landau levels below the Fermi level) influences effectively the magnitude of the direct current, then the height of the peak in our initial samples is  $W(1/N_{\nu} \approx 10^{-2})$ >  $10^4W(\pi/2)$ . The effective diameter for scattering by the dislocations, estimated from the data on the de Haas-van Alphen effect, which is close to the total scattering diameter, equals 1700 Å for  $\beta$  electrons and 6000 Å for  $\gamma$  electrons.

2. Change of anisotrophy of electron scattering in the course of uniaxial deformation. Typical experimental plots of the Dingle temperatures of  $\beta$  and  $\gamma$  electrons against the stress produced in sample are shown in Fig. 7. Two regions are clearly seen: 1) stress region where the Dingle temperatures and the oscillations amplitude are unchanged, corresponding to the Hooke strain region; 2) stress region where the Dingle temperatures increase (the oscillation amplitude decreases) with increasing load (plastic region) (Fig. 4). The am-



FIG. 5. Dependence of the relative change of the areas of the extremal electron orbits on the Fermi surface of aluminum in the third band:  $\nabla -\gamma$  orbits,  $\bigcirc -\beta$  orbits.

plitude decreases irreversibly during the deformation. The character of the change of the oscillation phase (Fig. 4) is different: first, the phase varies linearly with load at all the attained loads; second, the phase change is reversible, i.e., when the load is removed the oscillation phase returns to its previous value.

A plot of the relative change of the areas of the  $\beta$  and  $\gamma$  extremal sections of the Fermi surface of aluminum in a magnetic field parallel to the [110] axis, and at a load perpendicular to the magnetic field and parallel to the [111] axis, calculated from the change of the oscillation phase, is shown in Fig. 5. From the slopes of the lines on Fig. 5 we calculated the values of the parameters  $\mu$  (see formula (2)), namely  $\mu_{\rm g} = 90$  and  $\mu_{\gamma} = 60$  ( $\mu = 1$ in the free-electron model).

There is only one known publication<sup>[25]</sup> devoted to the effect of uniaxial deformation on the Fermi surface of aluminum, but the  $\beta$  orbits at B||[110] were not investigated there. The oscillation frequencies F coincided, for both types of oscillation, with the values obtained by Vol'skii<sup>(21)</sup>:  $F_{\gamma} = 2.8 \times 10^6$ G, and  $F_{\beta} = 5 \times 10^5$ G. The length L of the trajectory was calculated from the known area (which was calculated from the known frequency F) under the assumption that the  $\gamma$  trajectory is an equilateral triangle and the  $\beta$  trajectory is a circle.



FIG. 6. The Dingle temperatures X for the extremal  $\beta$  and  $\gamma$  orbits of the electrons on the Fermi surface of aluminum in the third zone. Different points on the plot correspond to different dislocation densities. The dash-dot line should be the locus of the points in the case of isotropic (in momentum space) scattering of the electrons by the dislocations. The dashed lines should be the loci of the points in accord with the Watts theory<sup>[7]</sup> in the limit of low and high dislocation densities. The circles mark the experimental results.



FIG. 7. Dingle temperatures of  $\gamma$  and  $\beta$  electrons in aluminum (curves 1 and 2, respectively) vs. uniaxial tension load in creep conditions at Dingle temperatures corresponding to the linear section of Fig. 6: •—first deformation,  $\bigcirc$ —deformation after heating for 5 hours to 300 K.

The change of the anisotropy with increasing dislocation density is best plotted with  $X_{\beta}$  and  $X_{\gamma}$  as coordinates. In the case of isotropic scattering (in momentum space) the experimental points should lie on a line making an angle 45° with the  $X_{\beta}$  and  $X_{\gamma}$  axes. This is the dashdot line in Fig. 6.

In the limit of low and high dislocation densities, the experimental points should lie, according to formulas (3), on the dashed lines of Fig. 6. The slopes of the plots correspond to the directly measured electronic parameters. The shaded region corresponds arbitrarily to the transition region where  $\xi_{\beta} \approx 1$  and  $\xi_{\gamma} \approx 1$ . The same figure shows the experimental points obtained in the course of the uniaxial deformation.

It is seen that the anisotropy, i.e., the ratio of the Dingle temperatures, does not remain constant when the dislocation density is increased, in agreement with the predictions of the theory, and changes from 3.5 to 2. The sign of the change of the anisotropy coincides with the prediction of the theory, but the experimental slope of the curve as  $D \rightarrow \infty$  is approximately double the theoretical. This disparity can be regarded as acceptable.

We conclude by noting the following. According to Watts<sup>[4]</sup> and Vinokur,<sup>[8]</sup> the Dingle temperature is proportional, in the limit of high dislocation densities, to the square root of the dislocation density,  $X \sim D^{1/2}$ , while according to classical concepts<sup>[26]</sup> the stress in face-centered metals deformed under creep conditions should have a similar dependence on the dislocation density,  $\sigma \sim D^{1/2}$ . Thus, in our experiments, at high dislocation density and far from the initial stage of plastic deformation, the following law should be satisfied:

 $X = A\sigma$ ,

i.e., a linear dependence of the Dingle factor on the

load. This is observed in experiment and is shown in Fig. 7.

It is of interest to investigate in greater detail, both theoretically and experimentally, the connection between the electronic characteristics and the parameters that describe the deformation of a metal under various conditions. This would lead to a better understanding, of the mechanisms whereby conduction electrons interact with a real crystal lattice, on the one hand, and of the metal-deformation mechanisms on the other.

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<sup>1)</sup>The difference between the coefficients of the proportionality of X and  $\tau^{-1}$  obtained by Bychkov<sup>[3]</sup> and by Dingle<sup>[2]</sup> leads to an insignificant redefinition of  $\tau^{-1}$ .

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