Cyclotron Resonance on Improved Specimens of Zinct

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Azbel'-Kaner cyclotron resonance has been studied at 35 Gc/sec and at temperature between 1.4 and 4.2°K in the three principal crystal planes of zinc. Fifteen distinct mass series were observed and their mass values determined as a function of the orientation of the magnetic field in the sample surface. The mass values of eight series are in general agreement with earlier work of Shaw, Sampath, and Eck, and of Henningsen. The angular range of observability of the limiting point resonance of the electrons on the spherical caps of the lens has been extended. In addition, seven new mass series have been observed in zinc for the first time and are assigned to orbits on the Fermi surface. Two of these mass series are in excellent agreement with the mass values given by the de Haas-van Alphen experiments of Joseph and Gordon. Magnetic breakdown between the first- and second-band hole surfaces has been observed, and an estimate of the energy gap between these bands near the point H in the Brillouin zone is given. A new technique for growing zinc samples in the desired orientation with optically flat unstrained surfaces was developed.

I. INTRODUCTION

`HE purpose of this paper is to present the results of an extensive experimental investigation of the Fermi surface of zinc by means of cyclotron resonance. Cyclotron-resonance experiments in zinc have been conducted by Shaw, Sampath, and Eck¹ (hereafter denoted by SSE), by Henningsen,² by Galt et al.,³ and by Naherezhnykh and Melnik,⁴ but only the papers by SSE and by Henningsen present extensive and systematic measurements of the cyclotron effective masses in zinc. The data of these two papers contain certain discrepancies which are resolved by the results presented in this paper. In addition, new data are presented here which extend the previously reported mass data in zinc.

The Fermi surface of zinc has been studied by many means and the most definitive mapping of the topology of the Fermi surface was done by Joseph and Gordon,⁵ and by Higgins, Marcus, and Witmore.⁶ They used the de Haas-van Alphen (dHvA) effect to map extremal cross sections of the Fermi surface, and for small crosssectional areas Joseph and Gordon measured the effective mass of the orbit from the temperature dependance of the amplitude of the dHvA oscillations. The mass values and orbit assignments in this investigation are compared to the dHvA measurements mentioned above.

Cyclotron effective masses for three principal crystal planes of zinc are presented and compared with the

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¹ Present address: Department of Physics, University of Washington, Seattle, Wash. ¹ M. P. Shaw, P. I. Sampath, and T. G. Eck, Phys. Rev. 142,

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 ² J. O. Henningsen, Phys. Status Solidi 22, 441 (1967).
³ J. K. Galt, F. R. Merrit, W. A. Yager, and H. W. Dail, Jr., Phys. Rev. Letters 2, 292 (1959).
⁴ V. P. Naberezhmykh and V. L. Melnick, Zh. Eksperim, i Teor. Fiz. 47, 873 (1964) [English transl.: Soviet Phys.—JETP 20, 583 (1965)].

⁵ A. S. Joseph and W. L. Gordon, Phys. Rev. **129**, 489 (1962). ⁶ R. J. Higgins, J. A. Marcus, and D. H. Whitmore, Phys. Rev. **137**, A1172 (1965).

results of previous mass studies^{1,2,5} in zinc. The results presented here are in general agreement with previous results, and seven newly observed mass series are presented and are assigned to orbits on the Fermi surface. Magnetic breakdown is observed between the first- and second-band hole surfaces. The results presented in this paper provide confirmation of several important features of the topology of the Fermi surface of zinc.

Observation of new cyclotron-resonance orbits in zinc was made possible by the development of a new technique for preparing specimens. Zinc single crystals of the desired orientations were grown with an optically flat surface.

II. EXPERIMENTAL

A. Measurement Techniques

The measurements reported in this paper were performed at temperatures between 1.4 and 4.2°K and at a microwave frequency of 35 Gc/sec using a standard microwave reflection spectrometer. The zinc sample formed one end wall of a cylindrical cavity resonating in the TE_{111} mode. The cavity was split, rotatable, and had a design similar to the one described by Spong and Kip.⁷ It was side-coupled to the broad face of the waveguide, and the zinc sample was lightly clamped with phosphor-bronze springs over the choke joint. The waveguide was suspended vertically between the pole faces of a 12-in. electromagnet which could be rotated about a vertical axis. With this arrangement, the horizontal magnetic field could be rotated relative to the sample surface and the sample could be rotated relative to a fixed combination of magnetic field and microwave polarization.

The dc magnetic field was modulated at an audio frequency and the klystron frequency was stabilized to the resonant frequency of the cavity. Thus, when the power absorbed by the cavity was a function of the magnetic field, the power reflected from the cavity

⁷ F. W. Spong and A. F. Kip, Phys. Rev. 137, A431 (1965).



FIG. 1. Graphite mold assembly for growing oriented zinc crystals with optically flat surfaces.

was modulated at the audio frequency, and the amplitude of the audio component was proportional to the derivative of the absorption with respect to the dc magnetic field, i.e., proportional to dR/dH, where Ris the real part of the surface impedance of the sample. The audio signal is detected, amplified, phase detected, integrated, and displayed on one pen of a two-pen chart recorder. In these experiments, the modulation was typically 1–4 Oe at 43 Hz.

The dc magnetic field was swept linearly with time and was measured by a Rawson rotating coil magnetic field probe whose output was displayed on the second pen of a two-pen chart recorder. Thus, the final recordings were plots of dR/dH versus **H**.

B. Sample Preparation

Prior to this investigation, cyclotron-resonance samples of zinc were prepared by cutting them from a single-crystal boule and the surfaces were prepared by chemical polishing. This method has two major disadvantages. First, cutting the boule introduces unwanted strains into the final sample. Second, and more critical, the polishing of the surface always produces a macroscopic "lemon peel" texture. This type of surface can produce splitting in some of the resonance signals, thereby giving the impression of an additional mass series and also causing an error in the measured mass values.⁸ To eliminate these "tip effects" caused by a wavy surface, all samples used in this investigation were grown with an optically flat surface of the desired crystallographic orientation. Data from the effects of tipping **H** out of the sample surface indicate that all samples were flat to within 6 sec of arc.

To produce a crystal with a plane-mirror surface, the zinc has to be grown on a polished flat surface, such as a Pyrex or quartz slide. However, since clean zinc welds to clean silica, a coating to prevent the zinc from sticking was developed. This coating consisted of a layer of carbon deposited on Pyrex slides by decomposing acetone vapor at 450° C. The method was to place clean Pyrex slides in a furnace tube at 450° C. Helium gas was then bubbled through an acetone bath, and the acetone laden helium gas was passed over the hot Pyrex slides for 5–8 h at a rate of 1 cm³/sec. The acetone decomposed slowly upon contact with the hot glass and produced a carbon coating which adhered tightly to the Pyrex. This coating completely prevented the zinc from sticking to the Pyrex slides.

The crystals were grown by the Bridgman technique in a graphite mold which was covered with two coated Pyrex slides as illustrated in Fig. 1. Samples grown in this manner had a typical residual-resistivity ratio of 22 000 and would exhibit from 30 to 50 subharmonics in a single cyclotron-resonance series. Eight different specimens were investigated, four with surfaces parallel to (1010), two parallel to (1120), and two parallel to (0001). The orientation of the normal to a sample surface was at worst within $\pm 2^{\circ}$ of the desired symmetry direction.

III. EXPERIMENTAL RESULTS

The Fermi surface of zinc according to Gibbons and Falicov⁹ is shown in Fig. 2. Because recent bandstructure calculations by Stark and Falicov¹⁰ show that there are no occupied electron states in the third and fourth bands centered around the point L in the Brillouin zone, and because there is no conclusive experimental evidence for the existence of occupied states at the point L, those sheets of the Fermi surface centered at L which were called the "butterfly-cigar" complex are omitted in Fig. 2. The various pieces of the Fermi surface have been labeled with descriptive names. The first band contains small pockets of holes, called caps, centered around the point H in the Brillouin zone. The second band contains a multiple connectedhole surface, the monster. The third band has two elec-

⁸ J. F. Koch, R. A. Stradling, and A. F. Kip, Phys. Rev. 133, A240 (1964).

⁹ D. F. Gibbons and L. M. Falicov, Phil. Mag. 8, 177 (1963).

¹⁰ R. W. Stark and L. M. Falicov, Phys. Rev. Letters 18, 795 (1967).



FIG. 2. Fermi surface of zinc according to Gibbons and Falicov. (a) First and second bands of holes (first-band "caps" are shown cross hatched). (b) Third band of electrons.

tron surfaces. One forms a surface, the lens, centered at the point Γ , and the other is an elongated ellipsoid structure, the needle, centered at the point K of the Brillouin zone. The Greek letters in Fig. 2 indicate orbits on the Fermi surface which are referred to in the following discussion.

The values of the cyclotron effective masses in zinc as a function of the orientation of the magnetic field held parallel to the specimen surface in the three principal crystal planes are presented in Fig. 3. Data were taken in the three planes with the microwave electric field \mathbf{E}_{rf} both parallel and perpendicular to the static magnetic field.

The four strongest resonances a, b, d, and e were studied in detail by SSE¹ and Henningsen² who interpreted them in terms of the Fermi surface of Gibbons and Falicov. Four other mass series were studied by SSE. They are the c_1 , f, i, and j branches of Fig. 3. We will limit comments on these eight mass series to new information or to confirmation of important features of the Fermi surface.

In addition to our study of the eight previously known mass branches, seven new resonances have been assigned to orbits on the Fermi surface. Typical accuracy in the mass values is $\pm 0.5\%$, with two exceptions. Branch *a* has typical uncertainties of $\pm 0.1\%$ because of a very high $\omega_{rf\tau}$ of about 35. Branch *n* has an accuracy from ± 3 to $\pm 1\%$ because the fundamental resonance occurs at less than 100 Oe for most of the angular range of observation and only three peaks could be observed.

The *a* resonance was the strongest signal present in the (1010) and (1120) surfaces and for most orientations had as many as 35 subharmonics as shown in Fig. 4. Branch *a* arises from the lens in the third band (ζ orbit of Fig. 2). Our measured mass values are in fair agreement with those of SSE and of Henningsen, except for an interval of 15° about the $\langle 0001 \rangle$ direction. We are convinced that our data are more accurate than those of previous workers, since we had a much flatter surface and much higher $\omega_{rf}\tau$. The data of branch *a* in Fig. 3 confirm the conclusion of SSE that the lens is







FIG. 4. Cyclotron-resonance signals in zinc for H parallel to $(11\overline{2}0)$ in a $(10\overline{1}0)$ surface with \mathbf{E}_{rf} perpendicular to H. The magnetic field scale is linear between each field value.

very nearly two spherical caps with the sharp corners slightly rounded. The *a* resonance was not observed in the (0001) surface by us, and it was noted by SSE that the signals from the a resonance were at least an order of magnitude weaker in the basal plane than in the other two planes. The much weaker signals or the absence of signals from the a resonance in the basal plane is explained in terms of the spherical cap model. For the $(10\overline{1}0)$ as $(11\overline{2}0)$ specimens, the electrons pass through the skin depth while on the flat top of the lens, while for the (0001) specimen, they are on the sharply rounded corners of the lens. As a result of this geometrical difference, electrons on the lens in the (0001) specimen spend much less time in the microwave skin depth and, therefore, have reduced coupling to the microwave field.

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One surprising result of our measurements was that the lens mass exhibited a measurable temperature dependence. A temperature study of the *a* resonance with **H** 14° from $\langle 0001 \rangle$ in a (1120) surface revealed a decrease in the cyclotron effective mass of 0.5% as the temperature was lowered from 4.2 to 1.1°K. The specimen studied had 52 subharmonics in the *a* resonance, and this large $\omega_{rf\tau}$ allowed a very accurate determination of the effective mass. Measurements are now underway to determine the temperature dependence of the lens effective mass between 4.2 and 15°K. The results of these experiments will be reported in a future publication.

The b resonance shown in Fig. 5, which is observable only with \mathbf{E}_{rf} parallel to \mathbf{H} , exhibits the peak splitting and inversions associated with a limiting-point resonance.^{7,11} Branch b is assigned to the electrons with orbits confined to the top and bottom of the lens. We have extended the angular range of observation of the b resonance to 24° from $\langle 0001 \rangle$, and at angles greater than 15° from the *c* axis, the limit-point mass begins to decrease rapidly. Since the NFE model predicts that the spherical caps subtend an angle of 35.5° from the center of the Fermi sphere, it is to be expected that the limit-point mass will decrease as the zone boundary is approached. All the above characteristics of the aand b resonances support the conclusion of SSE that the lens is shaped like two spherical caps with the corners rounded.

There is not much to add to the comments of SSE and Henningsen regarding the c_1 , d, and e resonances. We did not observe the c_1 resonance over the angular range of 20° to 50° from $\langle 0001 \rangle$, while SSE did. Our inability to resolve this resonance over the above angular range was probably due to the presence of the very strong a resonance. Our data on these resonances

¹¹ C. C. Grimes, A. F. Kip, F. W. Spong, R. A. Stradling, and P. Pincus, Phys. Rev. Letters 11, 455 (1963).



FIG. 5. Cyclotron-resonance signals in zinc for H parallel to $\langle 0001 \rangle$ in a (1010) surface with \mathbf{E}_{rf} parallel to H. The magnetic field scale is linear between each field value.

are in qualitative agreement with that of these investigators.

We could not assign branches f and i to a specific orbit on the Fermi surface of zinc, although both f and ihave the characteristics suitable for a multiple-zone orbit on the monster. The f resonance was tentatively assigned by SSE to the dog's bone orbit (λ of Fig. 2), but their mass value for this resonance is too small for the λ orbit. We have observed the λ orbit, and branch rin Fig. 3 is assigned to it.

Branch j, which SSE attributed to a lens orbit with nonextremal m^* , has been tentatively reassigned to an orbit on the inside ring of the monster. The *j* resonance, observed only with \mathbf{E}_{rf} perpendicular to \mathbf{H} , with a minimum m^* of $1.36m_0$ for **H** parallel to (0001), was observed in both the $(10\overline{1}0)$ and $(11\overline{2}0)$ surfaces to an angle of 12° from (0001) where the resonance abruptly vanished. The angular dependence of m^* and the polarization dependence of the signals are exactly the characteristics of a central orbit with a minimum mass. The orbit will vanish when H is at an angle from $\langle 0001 \rangle$, such that the plane of the orbit is tangent to the top and bottom of an opposite pair of horizontal arms of the monster. The limiting angle of 12° gives a minimum height for the horizontal arms of 0.19 Å^{-1} which is not consistent with the value of 0.13 Å^{-1} found by Higgins et al.⁶ Although our tentative assignment to the inside ring of the monster is open to question, we could find no other central orbit to which the jresonance could be assigned.

There are four mass branches reported by SSE, the g, h, l, and k branches, which we believe are due to tip effects. As is well known, tipping H out of the surface of the sample, or equivalently having a nonflat surface, will produce splitting in some of the resonance signals.⁸ These satellite peaks can easily be misinterpreted as a separate resonance series. Tipping can also cause some resonance series to be shifted in their field positions, thus, causing a shifted mass value for that series. In the (1010), SSE reported their h and k mass branches, neither of which were seen by Henningsen or by us. Tipping data in the angular range of the h branch taken by Henningsen and by us show that the *a* resonance exhibits splitting when the tip angle is as small as 10 min of arc. We also made tip measurements in the angular range of k and l branches and found that the aresonance shifted in mass value when \mathbf{E}_{rf} was parallel to H. Since, according to SSE, their samples were flat to 6 min of arc, we believe that these four mass branches reported by SSE are due to tipping effects.

We will now discuss seven newly observed mass series. Six of these have been assigned to the monster, and the seventh has been assigned to the needles in the third band.

The c_2 , c_3 , and c_4 resonances are seen only in the (1120) surface of zinc near the $\langle 10\overline{1}0\rangle$, and they are assigned to δ orbits on the monster which extend into 2, 3, and 4 zones. There are two classes of extended zone orbits. There are those which are centered about the symmetry line Γ -K and extend into an odd number of zones, and there are those which are centered about the symmetry line A-H and extend into an even number of zones. The *c* resonance which is confined to one zone vanishes at 13° from $\langle 10\overline{1}0 \rangle$, where the plane of the δ orbit becomes tangent to the saddle-shaped region formed at the six-arm junction near the point H in the Brillouin zone. The c_3 resonance then appears and vanishes at about 5° from $\langle 10\overline{1}0 \rangle$, where the plane of the orbit is tangent to two saddle-shaped regions separated by the height of three Brillouin zones. The even-zone orbits, the c_2 and c_4 resonances of Fig. 3, vanish when the plane of the orbit is tangent to two saddle-shaped regions separated by two and four times the height of the Brillouin zone. The c_2 resonance vanished at 7° from $\langle 10\overline{1}0 \rangle$, at which angle the c_4 resonance appeared. These resonances were observed only with \mathbf{E}_{rt} parallel to **H**. This is again expected, since the carriers on such extended orbits enter the skin depth of the $(11\overline{2}0)$ sample with most of their velocity parallel to **H**.

One surprising observation was that the c_2 , c_3 , and c_4 resonances suffered magnetic breakdown. For hexagonal closepacked metals, such as zinc, there is no energy gap to first order across the hexagonal face of the first Brillouin zone. However, an energy gap due to spinorbit splitting has been shown to exist between the first and second bands over most of the hexagonal face.¹² This means that the caps in the first band are split off from the diagonal arms of the monster by a small energy gap. For these small energy gaps, Cohen and Falicov¹³ and Blount¹⁴ have shown that magnetic breakdown across the gaps becomes probable when

$$\hbar\omega_c \ge E_g^2 / E_F, \qquad (1)$$

where ω_e is the cyclotron frequency, E_g is the energy gap, and E_F is the Fermi energy. For the two-zone orbit (c_2 resonance) which crosses the zone boundary twice, magnetic breakdown was almost complete at 5 kOe. From this approximate field value, we calculate an energy gap of 0.03 eV. This is in fair agreement with the calculated value of 0.028 eV at the point H of the Brillouin zone. Thus, we conclude, for magnetic fields greater than 5 kG, the caps of the first band are joined to the arms of the monster and the extended δ orbits no longer exist.

The *n* resonance is assigned to the α orbit on the needles in the third band. The resonance which is seen

with the same mass values in both the $(10\overline{1}0)$ and $(11\overline{2}0)$ surfaces has its fundamental at 84 Oe when **H** is parallel to $\langle 0001 \rangle$. The mass data are in good agreement with the dH-vA effect measurements of Joseph and Gordon.⁵ The value of m^*/m_0 when **H** is parallel to $\langle 0001 \rangle$ is 0.0073, compared to Joseph and Gordon's value of 0.0075. Our measurements provide the first direct values for the needle mass.

The *p* branch is seen centered about $\langle 0001 \rangle$ in both $(10\overline{1}0)$ and $(11\overline{2}0)$ surfaces with a minimum mass of $0.18m_0$. The observation that the mass is larger than that of the γ orbit and that it increases rapidly as **H** is rotated away from $\langle 0001 \rangle$, plus the correct polarization dependence for a central orbit, lead us to assign this branch to the orbit around the six-arm junction of the monster at the point *H* in the Brillouin zone, the μ orbit of Fig. 2.

The *r* resonance seen in the (0001) and (11 $\overline{2}$ 0) centered about $\langle 10\overline{1}0 \rangle$ is assigned to the dog's bone orbit of Higgins et al.⁶ (λ of Fig. 2) on the monster. The mass value along $\langle 10\overline{1}0 \rangle$ in (0001) is slightly less than twice that of the σ orbit in the same direction. This result is expected since the λ orbit consists of two σ orbits connected by the arcs along the horizontal arms of the monster. A fit of the mass values to a hyperbolic cylinder of revolution about the $\langle 11\overline{2}0 \rangle$ gives an asymptote, where the mass becomes infinite, of 18° from the $\langle 10\overline{1}0 \rangle$. The NFE model gives an angle of disappearance of 26° from $(10\overline{1}0)$.⁵ However, the NFE model overestimates the area of the horizontal arms of the monster by a factor of 10,⁵ and, when this is taken into account, the critical angle is reduced to 17° in agreement with fit to a hyperbolic cylinder. All of the above information supports the assignment of the r resonance to the dog's bone orbit.

The s resonance certainly arises from the β orbit of Fig. 2, the horizontal arms of the monster. The s branch is observed in all three principal crystal planes with a minimum m^* of $0.105m_0$. In the (0001), two branches crossing at $\langle 10\overline{1}0 \rangle$ are present, and this is expected since there are two pairs of horizontal arms, each pair 30° from $(10\overline{10})$. A fit of branch s to a hyperbolic cylinder of revolution about $\langle 11\overline{2}0 \rangle$ yields an asymptote of 66.5° from the axis of revolution. Joseph and Gordon have extracted effective masses for the β orbit from their dH-vA measurements, and, although their error is large (10%), their data are not in disagreement with that presented in Fig. 3. Their mass values indicate an asymptote somewhere around 70° from $\langle 10\overline{1}0 \rangle$, which is good agreement with our hyperbolic cylinder fit.

IV. SUMMARY

Four orbits in zinc previously observed by SSE and Henningsen and four orbits observed only by SSE were studied. The results of this study are in general agreement with the previous investigators. The

 ¹² M. H. Cohen and L. M. Falicov, Phys. Rev. Letters 5, 544 (1960).
¹³ M. H. Cohen and L. M. Falicov, Phys. Rev. Letters 7, 231

^{(1961).} ¹⁴ E. I. Blount, Phys. Rev. **126**, 1636 (1962).

angular range of observation of the limiting-point resonance on the lens and of the resonance from the diagonal arms of the monster were extended beyond that reported by previous workers. Four mass branches previously reported by SSE were not observed when **H** was parallel to the sample surface. Three of these resonances were reproduced in tip studies, and we are convinced that all four resonances are the result of tip effects.

Seven new cyclotron-resonance orbits in zinc have been observed. The orbits from the needles and from the horizontal arms of the monster were observed and their effective masses compared to the effective masses measured in the dHvA effect. Three orbits were observed which suffered from magnetic breakdown. The orbits were assigned to the Fermi surface, and the breakdown was interpreted as being between the firstand second-band hole surfaces. Based on the breakdown field, the energy gap between the first-band caps and the diagonal arms of the second-band monster was estimated to be 0.03 eV. This value is in agreement with the spin-orbit gap previously calculated by Cohen and Falicov.

The topologies and dimensions of the Fermi surface deduced from the mass anisotropies presented here are consistent with the results of other experimental techniques. The effective mass measurements reported in this paper support the current model of the Fermi surface of zinc.

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Enhancement of the Lattice Heat Capacity Due to Low-Frequency Resonance Modes in Dilute Aluminum-Silver Alloys*

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The lattice component of the specific heat of high-purity aluminum and of two aluminum alloys containing 0.5 and 0.95 at.% Ag, respectively, has been measured between 1.3 and 25°K. In the aluminum alloys, an enhancement of the lattice specific has heat been observed which can be explained in terms of low-frequency resonance modes associated with the heavy silver ions. A lattice-dynamical treatment of the heavy-mass defect in a light-host lattice is presented. The calculation is based on the aluminum density of states as obtained from neutron-scattering data, and the silver impurities are treated as mass defects only. The mass-defect calculation accounts for about 80% of the observed specific-heat enhancement. The values of the electronic specific-heat coefficient γ_0 and the Debye temperature Θ_D for pure aluminum are in agreement with previous data.

I. INTRODUCTION

THE phonon properties of a solid are affected appreciably by the presence of small concentrations of impurities which differ from the atoms of the host lattice in mass or in the interatomic force constants.¹ The introduction of isolated *heavy* impurities into a relatively light-host lattice causes a change in the phonon spectrum which is characterized by the existence of low-frequency resonance modes localized at the impurity site. Recently, the enhancement in the lattice-heat capacity at low temperatures caused by low-frequency resonance modes has been reported in a series of papers.²⁻⁴ Changes in the phonon spectrum due to small concentrations of very heavy or very light

^{*} Based in part on work performed under the auspices of the U. S. Atomic Energy Commission.

¹ A. A. Maradudin, in *Solid State Physics*, edited by F. Seitz and D. Turnbull (Academic Press Inc., New York, 1966), Vol. 18, p. 273.

² G. Kh. Panova and B. N. Samoilov, Zh. Eksperim. i Teor. Fiz. 49, 456 (1965) [English transl.: Soviet Phys.—JETP 22, 32D (1966)].

⁴ J. A. Cape, G. W. Lehman, W. V. Johnston, and R. E. DeWames, Phys. Rev. Letters 16, 892 (1966). ⁴ N. A. Chernoplekov, G. Kh. Panova, M. G. Zemlyanov, B. N.

⁴ N. A. Chernoplekov, G. Kh. Panova, M. G. Zemlyanov, B. N. Samoilov, and V. I. Kutaitsev, Phys. Status Solidi 20, 767 (1967).