Investigation of the critical field curves of lead at pressures up to 130 kbar and temperatures down to 0.1° K

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Moscow State University (Submitted April 28, 1975) Zh. Eksp. Teor. Fiz. 69, 1710–1722 (November 1975)

The effect of pressure up to 130 kbar on the critical magnetic field curves $H_c(T)$ of Pb is investigated in the temperature range from T_c to 0.1°K (where T_c is the superconducting transition temperature). It is shown that the deviation of the $H_c(T^2)$ curves from a straight line, which is characteristic of tight-binding superconductors (Pb, Hg at P = 0) and corresponds to the parabolic law $H_c(T) = H_c(0) [1 - (T/T_c)^2]$, reverses sign on increase of pressure. The change of the density N(0) of the electronic states at the Fermi surface due to compression is estimated on the basis of the experimental data and by using the thermodynamic relations. The density of states decreases by 27% with increase of pressure up to 130 kbar. The decrease of electron-phonon interaction due to compression seems to be the main cause of variation of N(0).

PACS numbers: 74.20.Gh, 74.50.Gz

The investigation of the critical magnetic fields H_c in a wide interval of pressures P and temperatures T is of particular interest for superconductors with strong electron-phonon interaction, such as lead. Upon compression, as follows from tunnel-effect measurements, ^[1,2] the ratio of the gap $\Delta(0)$ in the energy spectrum of superconducting lead to its superconducting transition temperature decreases with increasing pressure. On the basis of these data it is expected that lead should become a superconductor with weak binding at some pressure.

Measurements of the critical field curves $H_{\rm C}(T)$ under pressure make it possible to determine directly the pressure range in which this change takes place. The pressure dependence of the state density N(0) of normal electrons at the Fermi surface is the most valuable information that can be obtained from critical-field-curve measurements at different pressures. This information is significant both for the study of metal properties in the normal state and for the theory of strong-coupling superconductivity, which is undergoing intensive development of late. ^[3-6]

The critical magnetic field curves of lead were investigated by Garfinkel and Mapother ^[7] in the temperature range from T_c to 1°K and at pressures up to 0.65 kbar. We measured the critical fields of lead up to 130 kbar at temperatures down to 0.1°K.

EXPERIMENTAL PROCEDURE

1. Pressure Production and Measurement

Pressure was produced at room temperature in a high-pressure cell (Fig. 1) compressed between Bridgman anvils made of tungsten carbide with 3% Co binder (VK-3M). The cell consisted of two disks (2), $10-15 \mu$ thick, compacted from ultrafine powder of ferrous exide Fe₂O₃, of two guard rings (3), 20 μ thick, with 0.6 mm i.d. and 1.8 mm o.d., compacted from Fe₂O₃ powder in a special device, and of two 20- μ steatite disks (4) of 0.6 mm diameter. The specimen (5) in the form of a rectangular strip (0.6 mm long and 20 × 20 μ in cross section) was placed between the steatite discs. The steatite enclosing the specimen has high ductility and thus contributes to a very uniform distribution of the produced pressure.

We investigated lead samples of initial purity 99.99%, subjected to multiple zone cleaning in a quartz ampule. The superconducting transitions of the sample were revealed by the change of the resistance (at a current 1-5 mA through the sample). We investigated the central part of the sample, measuring 0.2×0.02 mm. The current and potential electrodes were four platinum strips (6 and 7) of approximate thickness 10 μ , connected, for greater reliability, with copper wires of 20 μ diameter, as shown in Fig. 1.

To produce pressures up to 130 kbar, we used an autonomous small booster.^[9] The positions of the anvils in the lower channel of the booster were fixed in such a way that the anvils could not be rotated. The use of the booster has made it possible to fix rigidly (automatically) the force compressing the anvil and developing during the course of freezing of water or acqueous solutions of undistilled alcohol in the upper channel of the booster, and to obtain a prescribed pressure on the investigated sample. All the parts of booster were made of "refined" beryllium bronze.

The pressure acting on the sample was determined from the plot of the superconducting transition temperature T_c against pressure for Pb.^[10,11] The pressure was determined accurate to $\pm 5\%$ (in consecutive compressions of the high-pressure cell, the accuracy of the relative measurement of the pressure was much higher).

2. The Cryostat

The measurements were made in the temperature interval $0.1-7.2^{\circ}$ K. The booster, connected to a pellet of iron-ammonium alum through a copper cold finger ~15 cm long, was suspended on a Staybrite wire inside a glass ampule evacuated by a carbon pump.^[9] The adiabatic demagnetization of the salt was carried out with a superconducting solenoid. It was shown earlier ^[12] that at T = 0.1° K the maximum time required for equilibrium to set in between the salt and the sample does not exceed 30 min. The heating of the salt from 0.1 to 0.6° K took ~4 hours, so that measurements could be made at



FIG. 1. Construction of the highpressure cell: 1-anvils, $2-Fe_2O_3$ disks, 3-guard rings, 4-steatite washers, 5sample, 6, 7-potential and current electrodes.

practically constant temperature. Temperatures above 4.2° K were obtained by heating the booster (vacuuminsulated from the helium bath) by an alternating magnetic field of 50-Hz frequency, produced by a copper solenoid placed in the vessel of a dewar with liquid nitrogen. The temperature was measured with an Allen-Bradley carbon thermometer secured in slots of the booster opposite the sample. This system ensured high stability of the obtained temperature and an accuracy up to 0.01°K. in its measurement.

3. Production and Calibration of Magnetic Field

The magnetic field acting on the sample, parallel to the planes of the anvils, was produced by a system of superconducting Helmholtz coils with constant 126 Oe/A. The frozen-in flux did not exceed 0.5% of the field introduced beforehand. Measurements in a magnetic field are complicated by the fact that the anvils, whose magnetic properties are governed by the cobalt impurities, become magnetized when the field is introduced, as do the parts of the Fe_2O_3 cell. This leads, on the one hand, to a change in the value of the field in the interior of the sample and to need for special calibration. On the other hand, after the field is turned off, a remanent magnetic moment is retained in the anvils and its direction, if the booster is freely suspended (as a result of its possible rotation about the vertical axis after the field is turned off) can be arbitrary relative to the direction of the measuring field. To prevent rotation, the booster was centered in the ampule with the aid of a special device. [9] This measure, as well as the rigid fastening of the anvils in the booster, made it possible to orient the investigated lead sample in such a way that the magnetic field was always directed along the sample, accurate to several degrees. The rigid fastening made it also possible to mount the sample at the center of the Helmholtz coils with accuracy ± 1 mm both in height and in radius, in the region where the magnetic field was most uniform.

The calibration curves of the field H acting on the sample against the external field H* were determined at different values of the residual magnetization of the anvils with the aid of superconducting and bismuth magnetic-field meters placed in the cell at the point of sample location. The calibration was carried out on several pairs of anvils, from the same batch. The obtained $H(H^*)$ plots, with H up to 1 kOe, are straight lines with almost equal slopes, which are shifted with increasing prior magnetization of the anvils. The value of H_c for each temperature was determined from the corresponding calibration curve with allowance for the preliminary

introduced field. The error in the determination of the magnetic field acting on the sample, including the error due to the possible difference in the Co concentration when different anvils are used and due to small difference in the cell heights, did not exceed, taking the calibration into account, 2%. As an additional control on the error introduced by the magnetic anvils, we plotted the destruction of the superconductivity of lead by a magnetic field at $T = 4.20^{\circ}$ K and at pressures 8, 41, and 61 kbar in ceramic anvils with guard rings of pyrophilite, which has no ferromagnetism. The values of H_c obtained with ceramic and magnetic anvils coincide, when the calibration is taken into account, within the limits of the indicated accuracy (Fig. 2).

The magnetic field produced at the location of the sample, at a maximum field in the superconducting solenoid intended for adiabatic demagnetization, amounted to ~8 Oe; with the field removed when all the measurements were made, the magnetic field at the sample was ~0.4 Oe, and introduced practically no additional uncertainty in the determination of the magnetic field acting on the sample. Four measurement runs were performed on two pairs of anvils. In each run, the measurements were made with successive compression of the same high-pressure cell; this increased the relative accuracy in the determination of the magnetic field to 1.5%.

MEASUREMENT RESULTS

1. Plots of Superconducting Transitions in a Zero Magnetic Field

Figure 3 shows plots of the transitions of lead into the superconducting state, obtained in a zero magnetic field at various pressures up to 130 kbar. The results of four measurement runs are given (runs 1 and 2 were made on anvil pairs different from those used in runs 3 and 4). The curves are numbered for each measurement run in chronological sequence. The temperature T_c was determined from the point of intersection of the linear part of the transition and the horizontal line corresponding to the constant value of the resistance r_0 prior to the start of the superconducting transition of the lead. The width of the linear section of the transition is determined by the inhomogeneity of the pressure in the cell, a distinguishing feature of the operating condition of the cell described above.

2. Plots of Superconducting Transitions in a Magnetic Field

By way of example, Fig. 4 shows some curves of the destruction of the superconductivity of Pb by a magnetic field, for the fourth run of experiments, at various tem-

FIG. 2. Pressure dependence of the critical magnetic field H_c of lead at $T = 4.2^{\circ}$ K: \bullet -measurements with ceramic anvils; \circ measurements with anvils made of VK-3M.





FIG. 4. Destruction of superconductivity of lead by a magnetic field at various temperatures and pressures (the abscissas represent the external magnetic field H*). a) P = 19 kbar; curves: 1-5.83 K, 2-5.65, 3-5.48, 4-5.34, 5-5.21, 6-5.08, 7-4.99, 8-4.87, 9-4.73, 10-4.57, 11-4.46, 12-4.21, 13-3.88, 14-3.60, 15-3.40, 16-3.21, 17-3.04, 18-2.86, 19-2.66, 20-2.47, 21-2.34, 22–2.18, 23–1.84, 24–0.14. b) P = 72 kbar, curves: 1-4.61 K, 2-4.53, 3-4.44, 4-4.35, 5-4.29, 6-4.22, 7-3.90, 8-3.60, 9-3.39, 10-3.20, 11-3.05, 12-2.87, 13-2.66, 14-2.45, 15-2.32, 16-2.20, 17-2.09, 18-1.91, 19-1.77, 20-0.15. c) P = 129 kbar, curves: 1-3.59 K, 2-3.39, 3-3.21, 4-3.06, 5-2.86, 6-2.64, 7-2.49, 8-2.35, 9-2.19, 10-2.09, 11-1.98, 12-1.84, 13-0.18.



H.^{*}Oe

peratures and pressures (a-19 kbar, b-72 kbar,c-129 kbar). Attention is called to the peculiarities of the presented curves.

At high temperatures (near T_c) the curves of the superconducting transitions in a magnetic field are very abrupt. The character of the curves changes with decreasing temperature depending on the value of the applied pressure. At pressures below ~ 30 kbar the height of the initial (in the region of weaker fields) steep part of the transitions decreases in magnitude with decreasing temperature, and its slope increases (Fig. 4a). At the same time, the smearing of the curves in the region of stronger fields increases strongly at the end of the superconducting transition.

At pressures above ~ 30 kbar (Figs. 4b and 4c), the slopes of the initial sections of the curves remains practically unchanged, down to the lowest temperatures. The initial sections increase with increasing pressure. A corresponding narrowing takes place in the interval of the fields that determine the degree of smearing of the ends of the superconducting-transition curves. The indicated singularities of the curves are typical of lead and are not connected with the procedure for producing high

pressures, since the superconducting-transition curves of lead, in analogous measurements, are steep and retain their shape in the entire range of temperatures and pressures.

The change in the character of the destruction of the superconductivity of lead by a magnetic field with decreasing temperature and pressure is apparently a consequence of the change of the Ginzburg-Landau parameter $\kappa(\mathbf{T}) = \lambda_0(\mathbf{T})/\xi(\mathbf{T})$ ($\lambda_0(\mathbf{T})$ is the depth of penetration of the magnetic field, $\xi(T)$ is the coherence length), the value of which determines the field H_{c3} of the surface superconductivity [13]:

$H_{c3} = 2.40 \times H_{c}$

(H_c is the thermodynamic critical field). At $\kappa < 0.42$ we have $H_{c3} < H_c$ and the presence of surface superconductivity does not influence the form of the superconducting transition curves in the magnetic field. The situation changes at $\kappa > 0.42$, when H_{c3} becomes larger than H_c . The superconducting transition curves begin to be smeared out in this case in the region of strong magnetic fields.

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According to Smith et al., ^[14] the parameter κ of lead at P = 0 increases with decreasing temperature, from ~0.2 near T_c to 0.5-0.55 at 1°K, so that at T ~ 5.5°K we have $\kappa \approx 0.42$. Therefore at T < 5.5°K and P = 0, owing to the fact that the field H_{c3} of the surface superconductivity becomes stronger than the thermodynamic critical field H_c, the superconducting transitions become smeared out over the field interval from H_c to H_{c3}. ^[15]

It follows from general considerations that when the pressure is increased the parameter κ should decrease. An estimate of the change of κ upon compression can be based on the London formula for the penetration depth

$$\lambda_0(0) = (mc^2/4\pi n_s e^2)^3$$

and on the known expression for the coherence length, $\xi(0) \approx \hbar v_F/kT_c$, where m, e, n_s, and v_F are respectively the mass, charge, concentration, and velocity of the free electrons on the Fermi surface at T = 0, c is the speed of light, and k is Boltzmann's constant.

Upon compression, $\lambda_0(0)$ decreases slightly because of the increase of n_s , while $\xi(0)$ increases as a result of the decrease of T_c and the small increase of v_F , which leads to a decrease of $\kappa(0) = \lambda_0(0)/\xi(0)$; the decrease of κ with increasing pressure and the ensuing decrease of the field H_{c3} shorten the magnetic-field interval in which the superconductivity is destroyed, and make the transition curves more abrupt. However, the very fast narrowing of the curves in the pressure interval from 20 to 40 kbar cannot be fully attributed to this mechanism alone. It is quite possible that the very strong smearing of the curves at low pressure, due to the large values of H_{c3} , is also a consequence of the appreciable inhomogeneous stresses on the sample surfaces, causing an increase of κ in the surface layer, ^[16] and the rapid narrowing of the curves with increasing pressure is due to the closer approach of the sample compression to hydrostatic and accordingly to a decreased stress gradient on the surface.

The presence of surface superconductivity makes it impossible to determine H_a from the end of the linear section of the superconducting transition in a magnetic field. Therefore the critical magnetic field H_c was determined from the point where the linear part of the plots of the destruction of superconductivity by a magnetic field intersects the horizontal line corresponding to the constant value of the signal prior to the start of the destruction of the superconductivity. This definition of H_c corresponds to the definition of T_c and sufficiently accurate, owing to the negligibly small value of the sample demagnetizing factor. It is seen from Fig. 4 that the slope of the linear part of the plots of the destruction of the superconductivity by the magnetic field, particularly in the region of higher pressures, remains practically unchanged, and this serves as an experimental confirmation of the smallness of the demagnetizing factor.

3. Change of Critical Fields of Lead Upon Compression

The critical magnetic field plots $H_{C}(T)$ determined for the investigated samples by the method described above are shown in Fig. 5. The dashed curve at P = 0 was plotted from the data of Chanin and Torre, ^[17] who determined the critical fields of lead with maximum accuracy. The value of dH_{C}/dT at $T = T_{C}$, equal to 237 Oe/°K at P = 0, decreases monotonically to a value 146 Oe/°K at P = 129 kbar.

Some of the curves of Fig. 5 are shown (for the sake of clarity) in Fig. 6 in terms of the coordinate H_c and T^2 (the scale does not accommodate all the points obtained at infralow temperatures). The values of $H_{c0}(P)$ were determined by extrapolating to $T = 0^{\circ}$ K the values of $H_{c0}(T)$ measured at infralow temperatures. The values of H_{c0} determined in this manner differ by not more than ~ 1 Oe from the corresponding values of H_c at the temperature 0.1°K. The values of H_{c0} and T_c for different pressures are as follows:

P, kbar:	16	19	38	40	61	70	72	92	98	121	126	128	129
H _{co} , Oe:	661	659	557	559	484	457	445	399	384	343	330	322	318
T _c , K:	8.53	6.41	5.77	5.70	5.12	4.89	4.85	4.43	4.32	3.89	3.79	3.75	3.74

It is seen clearly from Fig. 6 that the deviation of the $H_c(T^2)$ plots from straight lines, which is typical of superconductors with tight binding (Pb or Hg at P = 0), and corresponding to the parabolic law $H_c(T) = H_c(0)[1 - (T/T_c)^2]$, reverses sign with increasing pressure. The character of this change is illustrated in Fig. 7, which shows the plots of $\Delta h = h - (1 - t^2)$ in the coordinates Δh and t^2 ($h = H_c/H_{c0}$ is the relative critical field and $t = T/T_c$ is the relative temperature). The



FIG. 5. Critical fields of lead at different pressures P (kbar): 1-0, 2-19, 3-40, 4-61, 5-70, 6-92, 7-98, 8-121, 9-129. Dashed curvedata of [¹⁷].







FIG. 7. Deviation of the plots of the critical magnetic fields of lead from a parabola at various pressures P (kbar): •-19, \checkmark -40, X-61, 0-98. Dashed curve-data of [¹⁷].

dashed curve in Fig. 7 was plotted from the data of [17] at P = 0.

If, as assumed, we approximate the critical-field curves by the polynomial

$$h(t)=1-\sum_{n=2}^{N}a_{n}t^{n},$$

than the coefficient

$$a_2 = 1 - \frac{\partial (\Delta h)}{\partial (t^2)} \Big|_{t}$$

i.e., its deviation from unity is determined by the slope of the $\Delta h(t^2)$ curves as $t \rightarrow 0$. For superconductors with weak binding we have in the BCS theory $a_2 \approx 1.07$. For lead at P = 0 (dashed curve in Fig. 7) $a_2 = 0.90$. The scatter of the experimental points at $P \neq 0$, which is connected with the error in the determination of H_c , leads to a considerable inaccuracy in the determination of a_2 at each fixed pressure. Nevertheless, a large number of the measured $H_{c}(T, P)$ curves (the majority of which are not shown in Fig. 7) allows us to draw definite conclusions both concerning the change of the shapes of the $\Delta h(t^2)$ curves and concerning the change of the coefficient a_2 upon compression. Under the influence of the pressure, the coefficient a_2 increases, goes through unity at $P \approx 40$ kbar and reaches a value ~1.07 at P = 130 to 130 kbar. At sufficiently high pressures the coefficient a2 remains constant within the limits of measurement accuracy, and the $\Delta h(t^2)$ curves become similar in shape to the curves for superconductors with weak binding.

A theoretical estimate of the change of the coefficient a_2 at a pressure 24 kbar, corresponding to a 5% change of the volume v of the sample, is given in the papers of Carbotte and Vashishta, ^[5,6] who obtained d ln $a_2/d \ln v \approx -2$. According to our data, d ln $a_2/d \ln v \approx -(1.6 \pm 0.3)$ at low pressures (we used in the calculation White's value ^[18] of the compressibility of lead at low temperatures).

4. Change of the Density of the Electron States N(0) Upon Compression

From the obtained data we can estimate the compression-induced change of the density N(0) of the electronic states on the Fermi surface. It is known that the value of N(0) per unit volume is given by the expression

$$N(0) = a_2 \frac{3}{4\pi^3 k^2} \frac{H_{\rm co}^2}{T_{\rm c}^2}.$$

Figure 8 shows the pressure dependence of the ratio $3H_{C0}^2/4\pi^3k^2T_c^2$. For each series of measurements, the points are marked by different symbols and are numbered in the order in which the experiments were per-

formed. The horizontal bars mark the inhomogeneity of the pressure in the cell, determined from the width of the superconducting transition, and the vertical bars at low and high pressures mark the possible error in the determination of H_{C0}^2/T_c^2 . The foregoing data seem to point to a monotonically slowing-down decrease of H_{C0}^2/T_c^2 as the pressure is increased to 130 kbar.

To determine the character of the dependence of the density of the electronic states on the pressure, it is necessary to take into account the change of the coefficient a_2 upon compression. The plot of N(0) against P, obtained in this manner, is shown dashed in Fig. 8. The error in the determination of N(0), with allowance for the possible inaccuracy in the determination of the coefficients $a_2(P)$, amounts to $\sim 5\%$. At P = 0, and apparently also in the pressure region >100 kbar, where lead is a weak-binding superconductor and a_2 = const, the accuracy in the determination of N(0) is somewhat better. The maximum change in the density of states at 130 kbar is $\sim 27\%$.

In addition to the method used in this paper to determine the density of states from the critical-field curves, there are a number of methods (based on the change of the volume on going from the superconducting to the normal state in a magnetic field, or based on the temperature dependence of the thermal-expansion coefficient) which make it possible to determine the electronic Grüneisen constant $\gamma_{\rm e}$, which characterizes the rate of change of N(0) at P = 0, namely $\gamma_{\rm e} = [d \ln N(0)/d \ln v]_{\rm P=0}$. The data obtained by these methods and our results are listed in the table.

It is of interest to determine the cause of the observed decrease of N(0) upon compression. It is known^[3, 20] that the change of the density N(0) of the electronic states on the Fermi surface is determined by the constant λ of the electron-phonon interaction and by the value N_{bs}(0) of the band density of states:

$$N(0) = N_{bs}(1+\lambda). \tag{1}$$

The change of λ upon compression should be particularly strong for tight-binding superconductors, which have a large λ at P = 0.

At the present time it is impossible to determine accurately the change of λ at very high pressures. To estimate them, say on the basis of the Éliashberg equation, ^[4] it is necessary to know how the phonon spectrum and the band density of states are altered by compression. It appears that the most accurate calculation of the change of λ under the influence of pressure in lead was carried out in ^[5, 21], where the values of λ at zero pressure and at 24 kbar were calculated. To estimate the change of λ at higher pressures, we can use McMillan's formula ^[22]

$$T_{c} = \frac{\Theta_{p}}{1.45} \exp\left\{-\frac{1.04(1+\lambda)}{\lambda-\mu^{*}(1+0.62\lambda)}\right\},$$
 (2)

where $\Theta_{\mathbf{D}}$ is the Debye temperature and μ^* is the

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°е	Method of determination						
5.0±0.9	From the critical field curves [7]						
2.1±0.8	From the change in volume in the transition from the superconducting to the normal state [¹⁹]						
0.7 ± 0.5	From the temperature dependence of the coefficient of thermal expansion [18]						
2.2 ± 0.5	Theoretical estimate [¹⁹]						
2.4 2.1	Present work						



FIG. 8. Dependence of the ratio H_{C0}^2/T_c^2 of lead on the pressure. The dashed curve is a plot of the density N(0) of the electronic states on the Fermi surface.

FIG. 9. Pressure dependence of the electron-phonon interaction constant λ , calculated from formula (2), and of the band density of states N_{bs}, calculated from formula (1). X-result of theoretical calculation in [¹].

Coulomb pseudopotential. Formula (2) was obtained for the phonon spectrum of niobium, and therefore describes the superconducting properties of other elements with limited accuracy. Nonetheless this formula, even if regarded as approximate, seems to account correctly for the functional connection between the superconducting transition temperature and the main parameters of the spectrum of the superconductor in the normal state. It can therefore be used to estimate the role of these parameters in the change of T_c of lead under the influence of pressure. To obtain this estimate, we use the known published data on the changes of the parameters Θ_D and μ^* upon compression.

It is customarily assumed that the parameter μ^* depends little on the pressure, and therefore we can assume it to be constant in first-order approximation, equal to 0.213 for lead. This value determines $T_{\rm C}$ of lead (formula (2)) at $P = 0, \, \Theta_{\rm D} = 105^\circ {\rm K}, \, {\rm and} \, \lambda$ = 1.519, $^{[5,\,21]}$ and agrees well in order of magnitude with the theoretical estimates. The change of $\Theta_{\rm D}$ with changing pressure is satisfactorily described by the formula

$$\Theta_{D}(P) = \Theta_{D}(0) (v_{0}/v_{P})^{\gamma_{g}}$$

with a Grüneisen coefficient $\gamma_g = 2.85$. [5, 21]

The dependence of λ on P calculated in this manner for lead is shown in Fig. 9. Naturally, this is only an estimate. The same figure shows the values of N_{bs} determined at different pressures from (1). Obviously, the accuracy of N_{bs} is insufficient to be able to state that it is increased or decreased by pressure. We recall that according to the free-electron model N_{bs} should increase like $v^{-1/3}$. It appears, however, that we can say

that this quantity varies little over the entire interval of employed pressures, and the main cause of the change of the density N(0) of the electronic states on the Fermi surface is in the case of lead the decrease of the electron-phonon interaction with increasing compression. The mass of the density of states on the Fermi surface should vary in analogy with the dependence of N(O) on P, namely, m^{*} = m_{bs}(1 + λ).

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Translated by J. G. Adashko 183