

Properties of Lead-Indium Alloys. II. Specific Heat*

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Specific-heat measurements have been made on a series of lead-indium alloys containing from 0 to 60 at. % indium. For each sample the data yield precise (within 1%) values for both the normal-state electronic specific-heat coefficient γ and the Debye temperature Θ_D . In addition, though with less precision, both the bulk thermodynamic critical field $H_c(0)$ and the first generalized Ginsburg-Landau parameter κ_1 have been obtained. These results, together with those previously obtained for the magnetic properties of the same system, provide a set of comprehensive data for bulk thermal and magnetic properties of Pb-In alloys. The data are examined in order to establish to what extent strong-coupling and electronic structure effects may need to be incorporated into existing theoretical treatments. A combination of both effects is found to provide a quantitative explanation of the electronic specific-heat changes observed on alloying.

I. INTRODUCTION

The lead-indium alloy system is the first of a series of alloy systems to be examined by us with the object of providing reliable and comprehensive experimental data to improve our understanding of superconducting alloy behavior. The present paper is one of a series reporting the results of our investigation. A previous paper¹ dealt primarily with magnetic properties. The basic set of data to be discussed here have been obtained from specific-heat measurements. Our main conclusions seemed to have significance outside the context of the Pb-In system and have been reported briefly elsewhere.² In this paper the experimental results are set out in detail. Debye temperatures, omitted from the brief report, are presented and discussed. Finally, all our data for the Pb-In system are collected together and our main conclusions summarized.

II. EXPERIMENTAL METHODS

A. Sample Preparation

The starting materials employed when preparing samples were 99.999% pure lead and indium. Each melt was quenched into a cylindrical ingot from which cylinders 2.5 cm diam by 2.5 cm long were machined. Chemical analysis was performed on at least two pieces cut from opposite ends of the ingot, but no solute concentration differences greater than 1% were encountered. A final chemical etch of the sample served to reduce impurities introduced on machining.

B. Cryostat

The Debye temperature of pure lead is only 104 K and is substantially reduced on alloying with indium.

For all our samples, then there is a large lattice contribution to the specific heat and measurements must be made over a temperature range extending to well below 1 K in order to separate out the electronic contribution. We therefore used a ³He cryostat which extended our working temperature range down to 0.5 K. The measurement itself employed the conventional technique of supplying an accurately known quantity of heat energy to the isolated sample and monitoring the resulting temperature rise.

However, careful precautions were necessary to minimize error, especially at the lowest temperatures. Some of the measures adopted in this work may be described by referring to the specimen chamber schematic (Fig. 1). This chamber, in its basic design, is similar to one described by Lou-Lounasmaa³ but includes the following modifications:

(i) The background heat leak to the chamber has been reduced by thermally anchoring tubes K and M to a separately pumped ⁴He pot at 1.2 K (not shown). Both the chamber and ⁴He pot are suspended inside an evacuated can surrounded by ⁴He at 4.2 K.

(ii) In order to measure the specific heat in the normal state it is necessary to apply magnetic fields of up to 8 kG to the samples. If the germanium resistance thermometer employed for temperature measurement is placed in such a field, troublesome magnetoresistance corrections can occur. This difficulty has been overcome by surrounding the thermometer with superconducting Nb₃Sn, using the same material as that employed in the shielding experiments of Benaroya and Morgensen.⁴ The sample itself still experiences essentially the full applied field, since the thermometer (N) and shield (O) are mounted well away from it on one end of a 5-cm-long copper post. The other end of the post

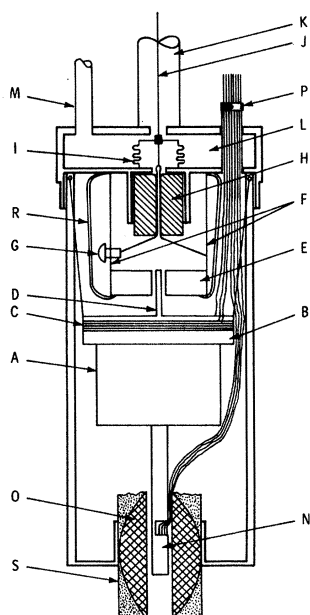


FIG. 1. Specimen chamber of cryostat: A, sample; B, heater cap; C, heater wires; D, gold ribbon; E, heat switch jaws; F, BeCu leaf springs; G, adjustment screw; H, quartz tube; I, bellows; J, heat switch wire; K, He^3 pumping line; L, He^3 pot; M, He^3 vapor pressure line; N, germanium thermometer; O, Nb_3Sn magnetic shield; P, glass metal seal; R, thermal contact with He^3 ; S, copper can for shield.

is screwed into the sample, Apiezon N grease being used for improved thermal contact. In operation, the magnetic field and its rate of increase were limited to maxima of 13 kG and 200 G/min, respectively. Under these circumstances it was found that no flux penetration occurred sufficient to affect the thermometer reading by more than the resolution of the measurement (± 0.5 mK). The thermometer calibration was divided into four overlapping ranges. Above 4 K it is traceable to the gas thermometer and vapor pressure calibration of Osborne *et al.*⁵ From 2.2 to 4 K and 0.7 to 2.2 K the vapor pressures of ^4He and ^3He , respectively, served as calibration standards. Between 0.4 and 0.7 K, the heat capacity of a piece of pure platinum was employed. The overlap consistency was to 1 mK except at 4 K where it was to 2 mK. All calibration data for the resistance R of the thermometer reading T between 0.4 and 8 K were least-squares fitted to the equation

$$T = \sum_{n=1}^2 A_n (\ln R)^{-n} + \sum_{n=0}^5 B_n R^{-n}. \quad (1)$$

(iii) A simple heat switch (E) similar to switches used by Manchester⁶ and Cochran *et al.*⁷ has been used. On opening, it introduces less than 10 erg of heat

energy to the sample. Cooling is achieved by pulling on the stainless steel wire (J), the linkage arrangement shown closing the jaws of the switch with a force of the order of 10^6 dyn. On releasing (J), the leaf spring (F) pulls open the switch, isolating the sample ready for measurement.

C. Measurement

Heat energy may be applied to the sample by passing current through a length of 0.0025-cm-diam Pt 9% W alloy wire. A measurement consists of electrically heating the sample for a predetermined period, usually of the order of 1 min, and observing the resultant change in sample temperature. Unavoidable extraneous heat inputs are allowed for in the usual way by monitoring the sample temperature between heating periods and extrapolating to the middle of the period to establish a drift-corrected change of sample temperature, ΔT . In addition, the measured heater power is corrected for the small effect of dissipation in the leads not wound directly onto the sample by adding half the dissipation in leads below the ^3He bath to the power supplied to the heater (C), a method proven by Neighbor⁸ to be quite accurate. Normal-state measurements were made in magnetic fields larger than $H_{c2}(0)$ where that field for each alloy was taken from our previous work.¹ The measured specific heat C_N was then least-squares fitted to the equation

$$C_N = \gamma T + \alpha T^3. \quad (2)$$

This expression was found to give a fit, to within experimental error, up to 1.7 K for pure lead and up to 1.2 K for the Pb-60%-In sample which has the lowest Θ_D . These limits correspond quite well with the expected range of validity of (2), namely, to temperatures below $\Theta_D/50$. Measurements were also made in zero magnetic field in order to establish C_s , the specific heat in the superconducting state. Using these experimentally determined values of $C_s(T)$ and $C_N(T)$, one can calculate the Gibbs potentials $G_s(0)$ and $G_N(0)$, and thence obtain $H_c(0)$, the thermodynamic critical field at zero temperature, from

$$H_c(0) = \{ 8\pi [G_N(0) - G_s(0)] \}^{1/2}. \quad (3)$$

III. RESULTS AND ACCURACY

The basic parameters established will now be presented, together with estimates of the accuracy with which they are known. The data are summarized in Table I which also records our previous results for convenience of reference.

A. Thermodynamic Critical Field $H_c(0)$

A value of $H_c(0)$ was found in two independent ways:

(i) Using the measured values of C_s and C_N to

TABLE I. Data for lead and some lead-indium alloys.

Parameter	Source	T_c (K)	γ_{exp} (mJ/mole K ²)	Θ_D (K)	$Z(0)$	$H_c(0)$ (Oe)	$H_c(0)$ (Oe)	$H_c(0)$ (Oe)	$\kappa_2(1)$	$\kappa_1(1)$	κ theor
Pb		7.19	3.000 \pm 0.002	104.11	2.49	...	803
Pb - 10 In		7.05	2.987 \pm 0.027	101.20	2.44	783	792	2.17	2.32	2.30	2.30
Pb - 20 In		6.91	2.973 \pm 0.021	97.47	2.30	774	776	3.50	3.66	3.77	3.77
Pb - 30 In		6.76	3.034 \pm 0.013	92.17	2.34	767	766	4.22	5.41	5.20	5.20
Pb - 40 In		6.57	2.993 \pm 0.009	87.17	2.29	738	749	4.52	5.71	5.79	5.79
Pb - 50 In		6.39	3.039 \pm 0.008	82.23	2.24	758	732	4.62	5.50	5.93	5.93
Pb - 60 In		6.21	3.095 \pm 0.028	78.65	2.19	...	716	4.15	5.15	5.32	5.32

compute the Gibbs potentials which were then inserted in Eq. (3), we obtained $H_c(0)$ for all alloys except Pb-60% In. [For that sample experimental difficulties prevented us from obtaining $C_s(T)$.] Although C_s and C_n are separately known to high precision, as will be discussed later, the difference between them is small enough to limit the experimental precision of $H_c(0)$ obtained in this way to $\pm 5\%$.

(ii) From the BCS theory we have

$$H_c^{\text{BCS}}(0) = \left(\frac{6}{\pi}\right)^{1/2} \frac{\Delta_0}{k} \gamma^{1/2}, \quad (4)$$

where Δ_0 is the gap parameter at $T=0$ K and k is Boltzmann's constant. Inserting the experimental values obtained here for γ and available values for Δ_0 , one obtains $H_c^{\text{BCS}}(0) = 875$ Oe for pure lead compared with the accepted experimental value of 803 Oe. It was shown by Swihart, Scalapino, and Wada⁹ that inclusion of strong coupling brings theory and experiment into quite close agreement. It has been shown experimentally that the parameter $2\Delta_0/kT_c$ changes less than 3% for Pb-In alloys over the entire range of primary solid solution, and also that the phonon spectra for the alloys are not drastically different from the spectrum for pure lead.¹⁰ These observations suggest that the strong-coupling effects in the alloys are likely to be similar to those in pure lead, and that one might estimate $H_c(0)$ for the alloys using the measured γ and T together with a simple scaling procedure, viz.,

$$H_c(0) = \frac{803}{875} H_c^{\text{BCS}}(0) = 803 \frac{T_c}{T_{c(\text{Pb})}} \left(\frac{\gamma}{\gamma_{\text{Pb}}}\right)^{1/2}. \quad (5)$$

Values of $H_c(0)$ obtained from this expression are listed in Table I and can be seen to be in reasonable agreement with those obtained from Eq. (3). The *experimental* precision enables one to evaluate Eq. (5) to 1% or better.

B. Ginzburg-Landau Parameter κ_1

In our previous paper¹ the behavior of the second generalized Ginzburg-Landau parameter was discussed in some detail. With the reliable values of $H_c(0)$ now available we now complete our study of the Ginzburg-Landau parameters by examining the behavior of κ_1 , defined by

$$\kappa_1(t) = H_{c2}(t)/\sqrt{2} H_c(t). \quad (6)$$

In general the temperature dependence of $H_c(t)$ may be expressed as

$$H_c(t) = H_c(0) [1 - t^2 + f(t)], \quad (7)$$

where $f(t)$ represents the departure from parabolicity of $H_c(t)$. To obtain $\kappa_1(1)$ we insert Eq. (7) into Eq. (6) and differentiate to obtain

$$\kappa_1(1) = -\frac{1}{\sqrt{2}} \left(\frac{dH_{c2}}{dt} \right)_{t=1} \left\{ H_c(0) \left[2 - \left(\frac{df}{dt} \right)_{t=1} \right] \right\}^{-1}. \quad (8)$$

The values of $(df/dt)_{t=1}$ for pure lead and indium are -0.07 and $+0.07$, respectively, and there is a good qualitative correlation in the pure element superconductors between the algebraic magnitude of this quantity and the coupling strength.¹¹ As already mentioned the electron tunneling evidence indicates that the coupling strength is approximately constant for the alloys. Hence, by adopting the pure lead value for $(df/dt)_{t=1}$ it is most unlikely that an error of more than a few percent is introduced into $\kappa_1(1)$. Using experimental values of $H_c(0)$ from Eq. (3) and $(dH_{c2}/dt)_{t=1}$ from Ref. 1 and setting $(df/dt)_{t=1} = -0.07$, the $\kappa_1(1)$ values shown in Table I were obtained. [An $H_c(0)$ value from Eq. (5) was used for the Pb-60%-In sample in the absence of an experimental $H_c(0)$ for this concentration.] From the above considerations we estimate a maximum error of $\pm 10\%$ for $\kappa_1(1)$ obtained in this way.

Turning to the temperature dependence of κ_1 , in our previous note we reported values of the ratio $\kappa_1(0.2)/\kappa_1(1)$, where $\kappa_1(1)$ was obtained from the (assumed) identity, $\kappa_1(1) = \kappa_2(1)$. However as can be seen from Table I, $\kappa_1(1)$ and $\kappa_2(1)$ show some deviation at the higher indium concentrations. Whatever this is due to, it is clear that it is incorrect to invoke the identity, as was previously done, and we return to Eqs. (6) and (7) to obtain the ratio in the form

$$\frac{\kappa_1(0.2)}{\kappa_1(1)} = 2h^*(0.2) \left[\frac{1 - \frac{1}{2}(df/dt)_{t=1}}{0.96 + f(0.2)} \right], \quad (9)$$

where

$$h^*(0.2) = -H_{c2}(0.2) / \left(\frac{dH_{c2}}{dt} \right)_{t=1}$$

is a parameter for which our direct experimental measurements coincide closely with available theory.¹² The term in brackets in Eq. (9) changes very little with coupling strength. From data in the literature, we calculate that for pure lead it is equal to 1.06, for pure indium 1.02, and in the BCS weak-coupling limit it takes the value 0.97. Furthermore, $h^*(0.2)$ changes very little with indium concentration; in fact, for all our alloys, $h^*(0.2) = 0.65 \pm 0.1$. Therefore, for all concentrations

$$\kappa_1(0.2)/\kappa_1(1) = 1.35 \pm 0.04. \quad (10)$$

The error limits include *both* the experimental uncertainties and the maximum uncertainty that we admit in the coupling strength, i. e., assuming simply that it lies somewhere between that for Pb and In.

C. Debye Temperatures

Values of the Debye temperature Θ_D are derived from the coefficient α in Eq. (2) in conjunction with the Debye model of the lattice specific heat. Results are set out in Table I. Errors, estimated from the mean-square deviation of a least-mean-squares fit to Eq. (2) are considerably less than 1% in all cases as can be seen from the table.

D. Electronic Specific-Heat Coefficient γ

Values of γ obtained from a least-mean-squares fit to Eq. (2) are set out in Table I, together with error estimated based on the deviations. There is remarkably little change in γ , less than 3%, across the entire alloy phase.

E. Other Collected Data

For convenience, some data from our previous paper¹ have also been presented in Table I. κ_{theor} is the theoretical value of the Ginzburg-Landau parameter calculated from the Gorkov equation

$$\kappa_{\text{theor}} = 2\sqrt{2} e H_c(t) \lambda_l^2 [\hbar \chi(\rho)]^{-1},$$

where e is the electronic charge, $H_c(t)$ is the bulk thermodynamic critical field of pure Pb, and λ_l is the London penetration depth in small fields,

$$\chi(\rho) = 0.949 \sum_0^{\infty} \frac{1}{(2n+1)^2(2n+1+\rho)}$$

and

$$\rho = 0.882 \xi_0 / l.$$

In the last expression, ξ_0 formally represents the coherence length for $l \rightarrow \infty$, and l is the average electron mean free path. Further details of this calculation are given in Ref. 1. $\kappa_2(1)$ is derived from the extrapolation of the $\kappa_2(t)$ data to $t=1$. $\kappa_2(t)$ was calculated directly from the initial and final slopes of the magnetization curves as reported in Ref. (1). T_c is the transition temperature and ρ_n the normal-state residual resistivity at 4.2 K. $Z_x(0)$ is the mass-renormalization parameter calculated for these alloys up to 45 at. % In by Wu¹³ using the data of Adler, Jackson, and Chandrasekhar.¹⁰ The result of that calculation could be expressed by

$$Z_x(0) = 2.49 - 0.005x, \quad (11)$$

where x is the atomic percent of In in the alloy. Equation (11) was used to obtain the results set out in Table I. It is not easy to assign an uncertainty to this estimate of $Z_x(0)$. However several previous investigations¹⁴⁻¹⁶ for pure lead have given values within 5% of that used in Eq. (11). Hence, it is thought that $Z_x(0)$ for our alloys is unlikely to be uncertain by more than this.

IV. DISCUSSION

A. Thermodynamic Critical Field $H_c(0)$

Referring to Table I, the two values of $H_c(0)$ obtained for each alloy agree to much better than the 5% error placed on the value obtained by integration of the specific-heat data. As can be seen from the table, $H_c(0)$ itself varies by less than 10% across the entire solid solution range.

B. First Ginzburg-Landau Parameter κ_1

The values of $\kappa_1(1)$ displayed in Table I evidently agree very well with κ_{theor} , obtained from the Ginzburg-Landau-Abrikosov-Gor'kov (GLAG) theory. This is in contrast with $\kappa_2(1)$ which shows a marked 25% deviation at higher concentrations. It is proper to recall that $\kappa_1(1)$ is shown only to $\pm 10\%$, $\kappa_2(1)$ to $\pm 5\%$, and κ_{theor} to $\pm 5\%$. Nonetheless, while not experimentally very clear cut, the 25% divergence between $\kappa_2(1)$ and $\kappa_1(1)$ does seem to suggest a breakdown of the widely assumed equality $\kappa_1(1) = \kappa_2(1)$. We note that the equality holds below 30 at. % In but breaks down above that concentration.

Turning to the temperature dependence of κ_1 , no theory is available which includes strong-coupling effects. If pure s -wave scattering is assumed, and if the BCS form for $H_c(t)$ is used, the predictions of Eilenberger¹⁷ for the ratio $\kappa_1(0, 2)/\kappa_1(1)$ reduce to those of Helfand and Werthamer¹² for the quantity h^* . Our direct experimental data are for h^* and they agree closely with the weak-coupling calculation of Helfand and Werthamer. If we further take reasonable values for $f(0, 2)$ and $(df/dt)_{t=1}$ in our alloys we get the estimate given by Eq. (10), viz., $\kappa_1(0, 2)/\kappa_1(1) = 1.35$. This differs somewhat from Eilenberger's result for the same ratio (~ 1.20) but the difference is small and, as remarked above, if BCS values are taken for $f(0, 2)$ and $(df/dt)_{t=1}$ his result is retrieved. Our conclusion is that the available weak-coupling theories for $\kappa_1(t)$ are both consistent with each other and essentially correct and that the strong-coupling corrections are likely to be small.

C. Debye Temperature

The values for Θ_D set out in Table I are plotted in Fig. 2 together with data obtained by other workers for pure lead¹⁸⁻²⁰ and lead-based systems.¹⁹⁻²² It can be seen that the initial rate of decrease can not be distinguished for In, Tl, and Bi and, furthermore, that the results for In and Tl as solutes are identical over our entire composition range. A somewhat similar general depression of Θ_D is observed for noble-metal alloys where only by introducing very light atoms such as Be or Al can one produce any increase of Θ_D . Zener²³ has given arguments which can be used to relate the noble-metal results to a change in elas-

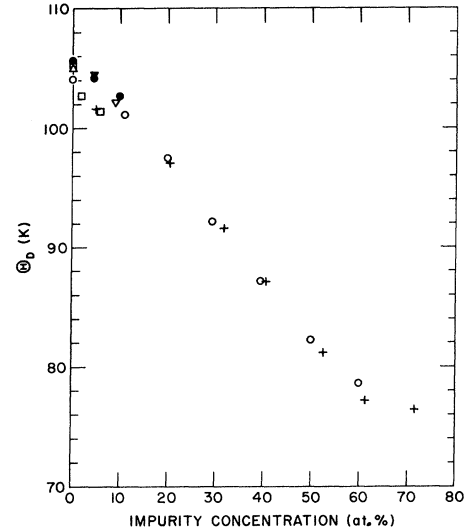


FIG. 2. Debye temperature Θ_D for lead and lead-based alloys: Δ Pure lead, Ref. 15; \circ Pn-In, this work; \square Pb-In, Ref. 17; \bullet Pb-Tl, Ref. 16; $+$ Pb-Tl, Refs. 18 and 19; ∇ Pb-Bi, Ref. 16.

tic moduli obtained from an elastic continuum model for the distortions round an impurity. However, the same arguments predict changes an order of magnitude too small to account for the data in lead alloys. Furthermore, the predicted effects depend on the square of the difference of the ionic radii and this factor is very considerably different for the three solutes. At this time it appears experimental information is available for only a few other basic physical parameters, but in nearly all cases the different solutes do produce significantly different effects (lattice spacings,²⁴ melting points,²⁵ and superconducting transit on temperature²⁶). In the case of residual electrical resistivity^{1,27} the data for In, Tl, and Bi scatter very little around a value of $\sim 0.8 \mu\Omega \text{ cm/at. \%}$, but one does not anticipate any particularly close connection between residual resistivity and the Debye temperature. Hence the identical values for Θ_D are quite surprising and although the coincidence is regarded as soundly established experimentally we are unable to offer any explanation for it at this time.

D. Electronic Specific-Heat Coefficients

The values set out in Table I are plotted against electron-atom ratio in Fig. 3. The band calculation by Anderson and Gold²⁸ for pure lead exhibits an increasing density of states with decreasing energy below the Fermi energy of lead. The rigid-band model can be applied directly to obtain a specific-heat coefficient for the alloys γ_R and the resulting variation of γ_R with \bar{z} (\bar{z} = electrons per atom) is shown normalized to the observed specific heat

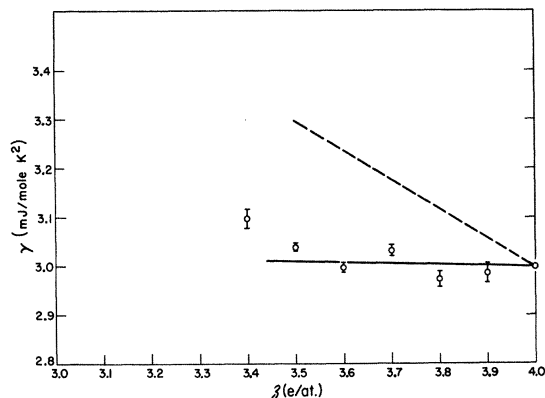


FIG. 3. Electronic specific-heat coefficient γ , plotted against electron/atom ratio \bar{z} for Pb-In alloys: Φ is the experimental values obtained in this work; dashed curve is γ_R , the "rigid-band" model prediction for γ (see text); solid curve is γ_R^* , the "rigid-band" model prediction, corrected for changes of electron-phonon coupling with composition (see text).

at $\bar{z} = 4$. The experimental data show an initial *decrease* as indium is added to lead whereas direct application of the rigid-band model gives an *increase*. However, there is a very strong electron-phonon coupling in these alloys and changes in its magnitude may be introduced into the rigid-band model by evaluating γ_R^* , where

$$\gamma_R^* = Z(0)\gamma_R / 2.49. \quad (12)$$

Values for $Z(0)$ were obtained from Table I and the resulting γ_R^* are plotted in Fig. 1 showing excellent agreement with the observed values for γ . Considering the sort of assumptions made by any rigid-band model, this agreement might be regarded as fortuitous were it not for the fact that Clune *et al.*¹⁹ have already demonstrated a similar agreement in the cases of both *PbBi* and *PbTl* alloys where the variations of γ differ in, respectively, sign and magnitude from the case of *Pb-In*. Our data lend convincing support to the quantitative adequacy of the modified rigid-band model and confirm the dominant role played by electronic structure and strong-coupling effects in the electronic specific heat of lead and its alloys.

E. Concluding Discussion

The over-all purpose of our investigation of the strong-coupling Pb-In alloy system has been to obtain comprehensive data on the magnetic and thermal properties and by comparing these with theory to establish the extent to which modifications

of the presently available weak-coupling spherical Fermi-surface treatments may be necessary.

Changes of the normal-state electronic specific-heat coefficient γ on alloying have provided perhaps the most striking indication of the general importance of strong-coupling and electronic structure effects for lead alloys. A simple calculation gives a quantitative account of the changes in γ and as has already been shown by Clune and Green¹⁹, this is also the case for *Pb-Tl* and *Pb-Bi* alloys. Some evidence has been presented on the basis of which we have speculated that a change in electronic structure occurs at roughly 30 at. % indium. The evidence consisted of small anomalies in the plots of T_c , normal-state resistivity and κ against composition, together with an apparent breakdown of the relation $\kappa_2(1) = \kappa_1(1)$. However experimental uncertainties are sufficient to leave some scope for drawing different smoothed lines through the data so that more precise measurements for very many more alloy compositions would be necessary to put the effect on a firm experimental foundation. Lattice spacing measurements²⁴ which should certainly reflect any such change do not seem to have been made over the composition range of interest.

Finally, turning to the possible effect of strong-coupling corrections on the temperature dependence of generalized Ginzburg-Landau parameters, our evidence is that any such corrections are at present less than experimental uncertainty and are likely to remain so for some time. The parameter

$$h^* = H_{c2}(t) \left[\left(\frac{dH_{c2}}{dt} \right)_{t=1} \right]^{-1}$$

is experimentally independent of indium concentration and a simple argument demonstrates that although it is not a direct observable, $\kappa_1(0.2)/\kappa_1(1.0)$ is therefore essentially independent of coupling strength and given correctly by present theory. Further, even though large discrepancies with theory are evident in our data¹ for $\kappa_2(0.2)/\kappa_2(1.0)$ very similar discrepancies seem to occur for weak-coupling alloys. The difficulty with κ_2 therefore appears to reflect a basic theoretical inadequacy which is unlikely to be resolved by invoking strong-coupling corrections.

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