rises *above* the GL prediction before ultimately going to zero at large fields and temperatures. As can be seen in Fig. 3, even at $\rho_c = 7.04$, $M'/H^{1/2}T$ falls off somewhat more slowly at large H/H_s than for the other curves. In the MT-type calculations, universal behavior appears to persist even into the very dirty limit.

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[27]D. Saint-James and P. G. DeGennes, Phys. Letters 7, 306 (1963).

[28]From Fig. 2 we see that $M'/H^{1/2}T$ falls to half the GL value at $b_c = 0.011$, which leads to the prediction $H_s = 0.11\ \Phi/2\pi\xi_0^2$.

[29]The range of fields over which data are shown for niobium and the alloys is determined at high fields by the high-field limit of reliable magnetometer operation (~ 300 Oe) and at low fields by broadening of the transition. Data judged to be affected by broadening are not shown in the figure. Inclusion of such data would have the effect of making the curve for each material rise rapidly above the indicated universal curve with a distinct change in slope at a point just below the lowest field data point shown for that material. The one exception is for In–16-at.% Tl where an unambiguous change in slope was not apparent and only a gradual departure from the universal curve was observed. The beginning of this departure can be seen in the point for this sample at $H/H_s \approx 1$. Since broadening may still be affecting the data in this one case, the indicated value of H_s for In–16-at.% Tl probably should be considered as an upper limit. (Suppose the observed magnetization were due solely to broadening. Then the fluctuations would have to have been suppressed at a lower field.)

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Temperature and Thickness Dependence of Critical Magnetic Fields in Lead Superconducting Films

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The parallel-field magnetic transitions of the pure Pb films have been determined from microwave absorption measurements as a function of the specimen thickness (700 to 15 000 Å) and of the temperature (3.2 °K to T_c). The parallel critical magnetic field H_{cF} values are in good agreement with the Ginzburg–Landau theory for $d < \sqrt{5}\lambda(t,d)$ and in the limit $d \gg \xi$; a general semiempirical expression for H_{cF} for all the values of the ratio $d/\lambda(t,d)$ has been determined. The order of the magnetic transition as a function of the specimen thickness and of the temperature has been studied. The surface-superconductivity properties as a function of the thickness have also been investigated and our results are in fairly good agreement with the Saint-James and de Gennes theory. From the data concerning the surface critical magnetic field H_{c3}, it was possible to compute the values of the penetration depth $\lambda(0,d)$ for any specimen thickness.

I. INTRODUCTION

The aim of this work is to make detailed studies on the magnetic behavior of pure lead films in a magnetic field parallel to the sample surface. The parallel magnetic critical field H_{cF} was measured as a function of temperature and specimen thickness. Lead, chosen for the present investigation, is an attractive material since it is a nearly local superconductor. Several measurements of the critical fields of lead films have been reported by a number of investigators,[1–5] whereas in these reports each of such measurements covers only a limited thickness or temperature range. Our measurements cover a range of specimen thickness varying from 700 to 15 000 Å and a temperature range between 3.2 °K and the critical temperature of the specimen T_c. This wide range enables us to study the phase-transition order by varying either the thickness or the temperature.

It is well known[6–9] that for thicknesses d, when $d < d_c = \sqrt{5}\,\lambda(t, d)$, a second-order transition is expected at H_{cF}, while for $d > d_c$, a first-order transition should occur at the critical field H_{cF}. Studies were also made to ascertain how thin-film behavior is replaced by the characteristic behavior of bulk specimen. For each sample thickness $d > \sqrt{5}\,\lambda(0, d)$ we determined a reduced critical temperature t_{d_c} so that for $t < t_{d_c}$ a first-order transition could be expected, while for $t > t_{d_c}$ a second-order transition should be observed.

In general, it was found that the temperature dependence of H_{cF} for thin films and for thicknesses $d \gg \xi(t)$, is in good agreement with the Ginzburg–Landau (GL) theory.[6,10] For films of intermediate thicknesses, no expression for H_{cF} yet exists; we found, however, a semiempirical functional expression to fit all the experimental data concerning H_{cF}.

In addition, we studied the changes in surface superconducting properties[11–15] as a function of the sample thickness. We determined the thickness dependence of the temperature $t_{H_{c3}}$ at which the surface superconducting sheath appears. The measured surface critical field H_{c3} is in good agreement with the prediction of the theory of Saint-James and de Gennes.[16]

II. EXPERIMENTAL PROCEDURE

The films used in this work were disk shaped and have been vacuum evaporated on cylindrical substrates (held at room temperature) made of electrolytic copper or crystalline quartz. The films were obtained by electron-gun evaporation at a pressure of about 10^{-7} Torr and at a rate of 50 Å/sec.

The substrates were washed in a saturated solution of detergent, rinsed in acetone, carbon tetrachloride, ethyl alcohol, and then air dried.

In order to avoid edge effects the films were prepared by evaporation through a mask which behaves as a stencil. Although trimming techniques have not been used, that edge effects are not present is apparent because the transitions in a magnetic field are very sharp (see following results). The lead used for our samples is 99.999% pure and was obtained from Ventron.

The film thickness was estimated by the evaporation rate and was determined by means of interferometric measurements.

The experiments, performed with a microwave-absorption spectrometer working in the K band (Strand. Labs. Corp. model No. 602 A/k) have been described in detail elsewhere.[17] The sample forms the plunger of a cylindrical cavity with TE_{011} excitation mode at a resonant frequency of 24 GHz. The surface-resistance derivative dR/dH has been measured as a function of the magnetic field parallel to the sample surface. The results were obtained by modulating the microwave power with a 200-Hz magnetic field and observing the ac component of the reflected power by means of a phase-sensitive technique.

All measurements were carried out on substrates made both of quartz and electrolytic copper. In general, if a superconductor comes into contact with a normal metal, a drastic change takes place in its own superconductive properties.[18–20] However, it was observed experimentally that the two situations (Pb/Cu or Pb/quartz) do not present significant differences, either in the values of the bulk critical fields or in the transition line shape. This is because there is always a layer of oxide on the copper surface which prevents a direct metallic contact between the superconducting film and the copper. Therefore, as observed also by Fisher *et al.*,[21] the boundary conditions required by the theory of Saint-James and de Gennes are satisfied.

III. EXPERIMENTAL RESULTS AND DISCUSSION

To present the experimental results the analyzed samples are divided according to the observed order of the phase transition at H_{cF}. Since, as previously mentioned, the quantity which determines the phase-transition order at H_{cF} is the critical thickness $d_c = \sqrt{5}\,\lambda(t, d)$, it is possible to divide the samples into three distinct thickness ranges: (a) thick-film region $d > d_c$, including the samples which show a first-order transition at H_{cF} over the whole temperature range covered in the measurements; (b) the thin-film region $d < d_c$, including the samples which show a second-order transition for every T; (c) the intermediate-thickness region. The samples belonging to this region show a second-order transition at temperatures near T_c and a first-order transition at lower temperatures.

A. Thick-Film Region $d > d_c$

The samples analyzed have the following thicknesses: 15 000, 7000, 6000 Å. Figure 1 shows a series of differential curves, which for simplicity are referred to as "superconductivity peaks," as a

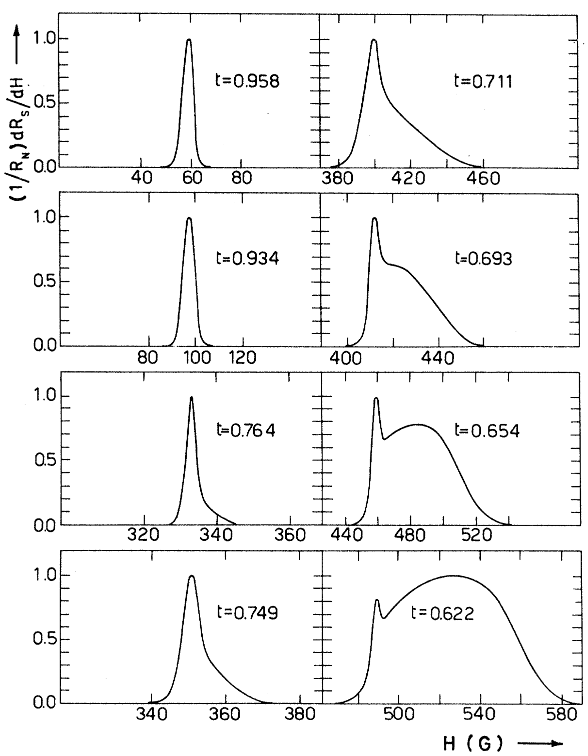

FIG. 1. Superconductivity peaks as a function of the magnetic field for several values of the temperature, for a sample of thickness $d = 15\,000$ Å. The curves have been normalized to the maximum value.

function of the magnetic field at different temperatures. Each transition appears as a sharp peak followed, under some conditions, by a bell-shaped structure. The H value for the first maximum (lowest field) corresponds to the bulk critical field H_{cF}. The measurements (Fig. 1) refer to a thickness $d = 15\,000$ Å and to a quartz substrate. The shape of the superconducting transition is symmetric in a temperature range near T_c. This is due to the presence of only a first-order transition at H_{cF}, which coincides (within the errors) with the thermodynamic critical field $H_c(t) = H_c(0)(1 - t^2)$ at this thickness. When temperature is decreased the line shape changes in correspondence to values of the magnetic field higher then H_{cF}.

This result is due to the existence of a surface superconducting sheath between H_{cF} and H_{c3} with thickness of about $\xi(t)$.

If the temperature is further decreased, the superconductivity peak clearly shows two components: the first one is again due to the first-order transition at H_{cF}, while the other one corresponds to a second-order transition at H_{c3}.

We need a criterion which allows the choice of a surface critical temperature t_{Hc3} defined in such a way that for $t > t_{Hc3}$, only bulk superconductivity occurs while for $t < t_{Hc3}$ the surface superconductivity is also present. For this purpose, the quantity $H' - H_{max}$ was measured as a function of the temperature, where H' is the magnetic field at which the superconductivity peaks reach zero and H_{max} is the magnetic field corresponding to the first maximum of the superconductivity peak. Let us assume that H' is the value of the magnetic field at which the signal amplitude is of the same order as the noise. In Fig. 2 the temperature dependence of the difference $H' - H_{max}$ at several thicknesses is shown. t_{Hc3} is defined as the first temperature at which $H' - H_{max}$ increases, compared with the constant value near T_c. Obviously, for $t < t_{Hc3}$, H' will assume the physical meaning of surface critical field H_{c3} (second-order transition). The value found for t_{Hc3} is in good agreement with that reported elsewhere.[22]

From the Saint-James and de Gennes theory,[16] we have, for $H_{c3}(t)$,

$$H_{c3}(t) = 1.695\,[4\pi\lambda^2(0,d)H_c^2(0)/\Phi_0]\,(1-t^2)/(1+t^2)\,, \tag{1}$$

where Φ_0 is the flux quantum and $\lambda(0, d)$ is the penetration depth at $T = 0$ °K.

A nonlinear least-squares curve-fitting technique has been used to fit our experimental results with Eq. (1). The only adjustable parameter is the penetration depth $\lambda(0, d)$; Table I summarizes all the values of $\lambda(0, d)$ obtained by this computation.

In Fig. 3 the temperature dependence of the critical field H_{cF} for the thickness $d = 7000$ Å is shown. The continuous curve is obtained by interpolating the experimental data with the following expression, valid in the limit $d \gg \xi(t)$[10]:

$$H_{cF}(t) = H_c(t)\left(1 + \alpha\,\frac{\lambda(t,d)}{d}\right)\,, \tag{2}$$

where α is a parameter introduced by Ginzburg.[10] In the fitting procedure α is assumed as a parameter; Table I shows also the α values obtained for all the samples.

The critical fields for the thicknesses $d = 7000$ and $d = 6000$ Å differ only by a few percent from those

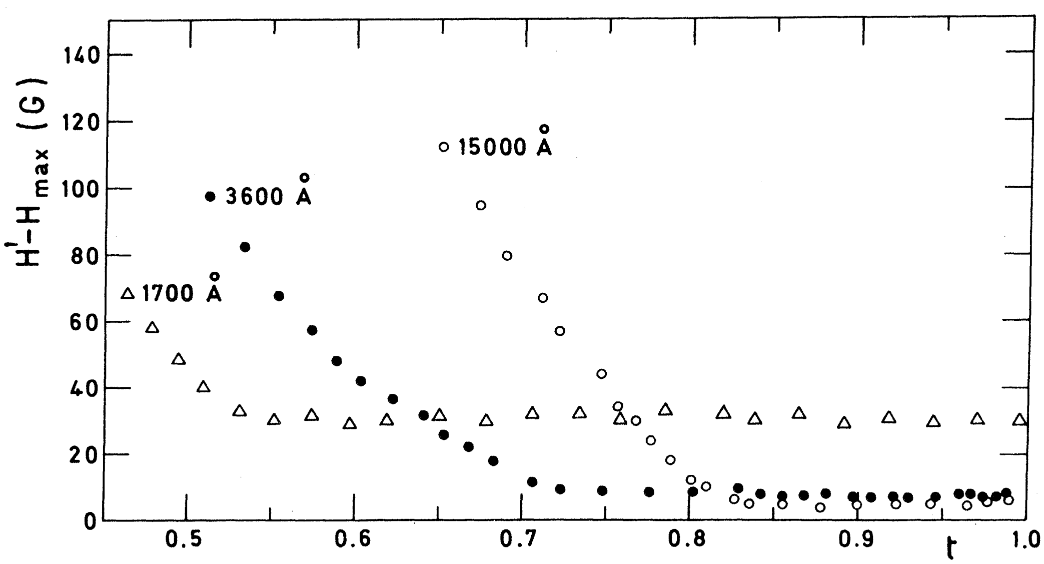

FIG. 2. Temperature dependence of $H' - H_{max}$ for several film thicknesses. H' is the magnetic field for which the superconductivity peaks go to zero; H_{max} is the field corresponding to the maximum of the superconductivity peak.

TABLE I. Summary of all values of $\lambda(0, d)$ obtained from Eq. (1).

d(Å)	$\lambda(0, d)$ Å	α	(Der.) $t_{H_{c3}}$	(Expt.) $t_{H_{c3}}$	(Der.) $k_{H_{c3}}$	(Expt.) $k_{H_{c3}}$
15000	448	...	0.845	0.829	0.42	0.43
7000	457	0.47	0.843	0.830	0.43	0.44
6000	456	0.47	0.830	0.817	0.44	0.45
4400	456	1.07	0.754	0.743	0.48	0.48
3600	452	1.05	0.718	0.709	0.49	0.50
3000	448	1.28	0.644	0.640	0.51	0.51
2800	450	1.31	0.638	0.636	0.52	0.53
2500	468	1.75	0.620	0.618	0.56	0.56
2200	477	1.88	0.593	0.587	0.60	0.60
1700	488	1.95	0.534	0.530	0.66	0.65
1000	520	...	...	...	...	...
700	537	...	...	...	...	...

found for the thickness $d = 15\,000$ Å; that is, these samples show the typical behavior of the semi-infinite superconductors.

B. Thin-Film Region $d < d_c$

The samples analyzed have the following thicknesses: 1000 and 700 Å. These samples, in agreement with the GL theory, show a second-order transition for each t. For these thicknesses, which are comparable with the coherence length ξ_0, it is not possible to distinguish surface from bulk properties and, therefore, we cannot define a surface critical field.

The characteristics of the phase transition are clearly observed in the family of curves in Fig. 4 for the thickness $d = 700$ Å. The curves are seen to be asymmetrical; this asymmetry is measured by means of the quantity $A = (\Delta H_{1/2})_L / (\Delta H_{1/2})_R$, where $(\Delta H_{1/2})_{L,R}$ is the half-width at the half-height of the superconductivity peak. For first-order transitions $A = 1$, while for second-order transitions $A > 1$.

In Fig. 3 the temperature depencence of H_{cF} is shown. The value of the magnetic field at which the superconductivity peak drops to zero for second-order transitions is compared with the following expression, using $\lambda(0, d)$ as a parameter:

$$H_{cF}(t) = \sqrt{24}\, H_c(0)\, \frac{\lambda(0, d)}{d} \left(\frac{1 - t^2}{1 + t^2}\right)^{1/2} . \tag{3}$$

The values obtained for $\lambda(0, d)$ are listed in Table I.

C. Intermediate-Thickness Region

The analyzed samples belonging to this range have the following thicknesses: 4400, 3600, 3000, 2800, 2500, 2200, and 1700 Å.

Figure 5 shows an example of the superconductivity peak for a thickness $d = 2500$ Å as a function of the magnetic field for several temperature values; the line shape is much more complex than that observed in the two previously considered

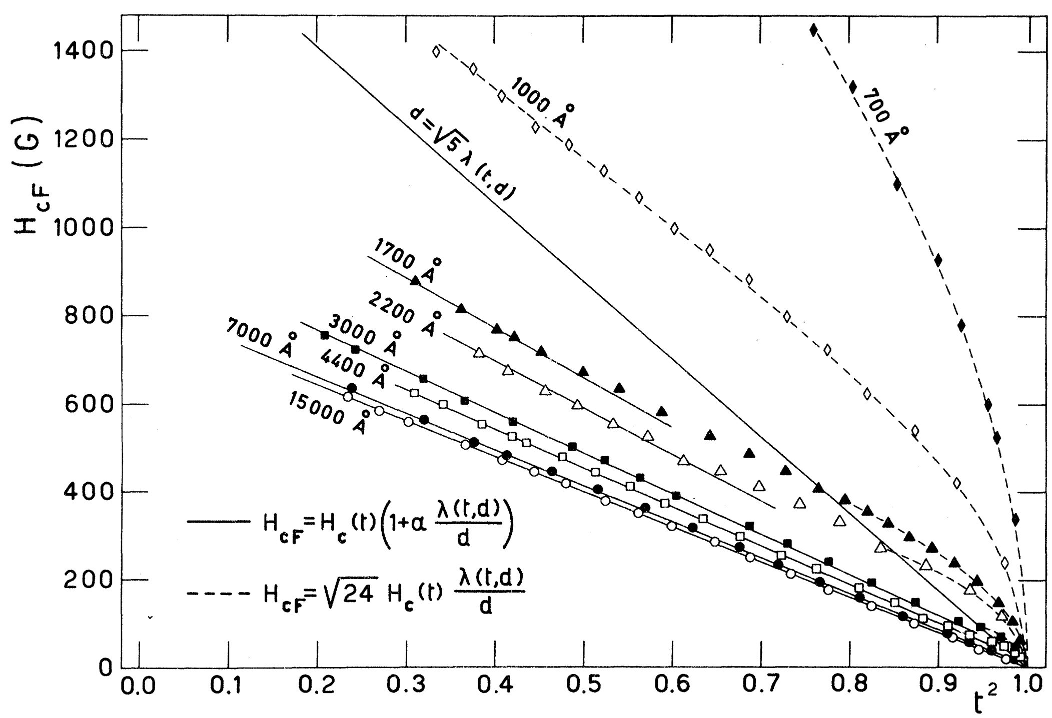

FIG. 3. Parallel critical field H_{cF} as a function of t^2 for several film thicknesses.

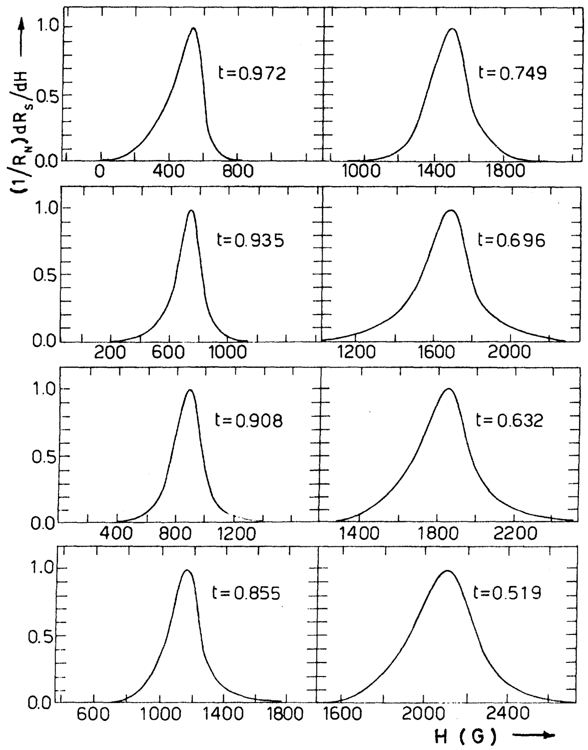

FIG. 4. Superconductivity peaks as a function of the magnetic field for several values of the temperature, for a specimen of thickness $d=700$ Å. The curves have been normalized.

ranges.

Indeed, for these samples, by varying t it is possible to pass from the condition of thin film ($d<d_c$) to that of thick film ($d>d_c$). This thin-film bulk transition is continuous.

If t_{d_c} is the temperature at which

$$d=d_c=\sqrt{5}\,\lambda(0,\,d)/(1-t^4)^{1/2}\ , \tag{4}$$

from Eq. (3), for $d=d_c$, we get

$$H_{cF}=2.19H_c(t)\ . \tag{5}$$

In Fig. 3 the line joining the points $H_{cF}=2.19H_c$ is represented. Thus, it is possible to determine experimentally the temperature (denoted here as t'_{d_c}) at which condition (5) holds.

It must be remembered that the values t_{d_c} and t'_{d_c} are derived from two sets of independent measurements; in fact, t_{d_c} is calculated using the values of $\lambda(0,\,d)$ obtained by a fitting of $H_{c3}(t)$, while t'_{d_c} is derived using the experimental values of $H_{cF}(t)$.

From the curve shape it is possible to find the temperature t''_{d_c} at which we go from a second-order transition to a first-order transition.

For this purpose Fig. 6 shows the asymmetry degree A of our differential curves as a function of the temperature; these curves refer to the thicknesses 1700, 2500, and 2800 Å.

As can be seen from Fig. 5, the curves are asymmetrical near T_c and thus $A>1$, while they become symmetrical further away from T_c and thus $A=1$. As previously seen, this behavior is typical of second- and first-order transitions, respectively.

The temperature at which A shifts from the constant value is assumed to be t''_{d_c}. The temperatures t_{d_c}, t'_{d_c}, and t''_{d_c} derived by different methods for all the samples analyzed are listed in Table II.

These temperatures are in good agreement among themselves. In the family of curves presented in Fig. 5, it can be seen that by decreasing the temperature further, the superconductivity peak is modified at $H>H_{cF}$ because of the surface superconductivity.

The critical temperature $t_{H_{c3}}$ was determined using the criteria described in the limit $d>d_c$. The values obtained for all the samples analyzed are shown in Table I.

As far as the analysis of the temperature dependence of H_{cF} is concerned, for $d<d_c$ the fitting of the experimental results was performed by using Eq. (3).

For $d>d_c$ and $d\gg\xi$, Eq. (2) was used, which

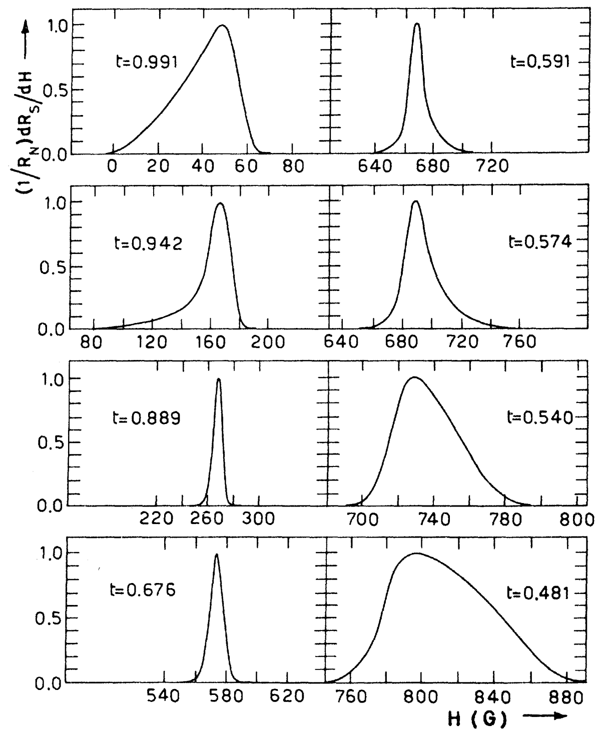

FIG. 5. Superconductivity peaks as a function of the magnetic field for several values of the temperature, for a specimen of thickness $d=2500$ Å. The curves have been normalized.

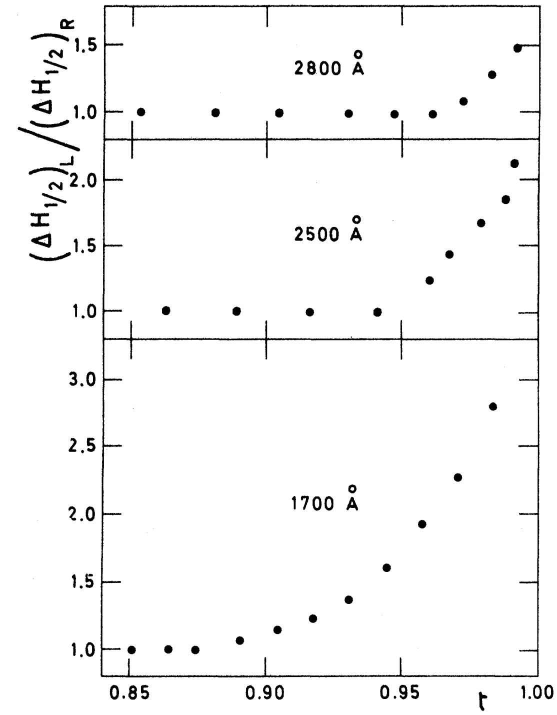

FIG. 6. Temperature dependence of the asymmetry degree $A = (\Delta H_{1/2})_L / (\Delta H_{1/2})_R$ for the thicknesses 1700, 2500, and 2800 Å.

was found to hold for thicknesses of $d = 4400$ and $d = 3600$ Å. The fitting (continuous line in Fig. 3) was carried out by a method of nonlinear least squares using α as parameter; the values obtained

TABLE II. Analysis of temperatures t_{d_c}, t'_{d_c}, and t''_{d_c} derived by different methods for all samples.

d(Å)	t_{d_c}	t'_{d_c}	t''_{d_c}
15000	...	...	...
7000	0.995	...	...
6000	0.993	...	...
4400	0.986	0.986	0.987
3600	0.980	0.978	0.975
3000	0.971	0.972	0.970
2800	0.966	0.960	0.965
2500	0.953	0.948	0.945
2200	0.935	0.927	0.930
1700	0.876	0.880	0.880

of the order of one, agree well with those predicted by Ginzburg[10] and are listed in Table I.

The GL theory does not supply a valid expression for $d \geq \xi$, however, we obtained a good agreement using Eq. (2) for samples 3000, 2800, 2500, 2200, and 1700 Å thick at temperatures far from T_c, when the values of α are larger than those predicted by Ginzburg in the limit $d \gg \xi$.

The problem is then to find a semiempirical functional expression to fit all the data relative to H_{cF} for any thickness, taking into account the asymptotic expressions (2) and (3). Figure 7 shows the ratio $H_{cF}(t)/H_c(t)$ as a function of the ratio $d/\lambda(t,d)$. The fitting was done by a nonlinear combination of the expressions (2) and (3) valid within the limits $d < d_c$ and $d \gg \xi$

$$\frac{H_{cF}(t)}{H_c(t)} = \left[1 - F\left(\frac{d}{\lambda(t,d)}\right)\right] \sqrt{24}\, \frac{\lambda(t,d)}{d}$$

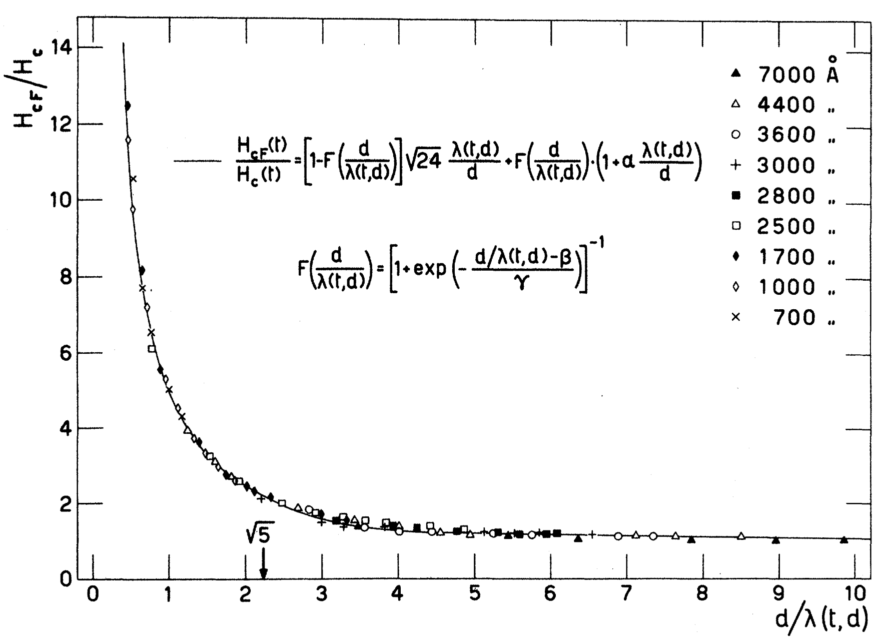

FIG. 7. $H_{cF}(t)/H_c(t)$ as a function of $d/\lambda\,(t,d)$.

$$+F\left(\frac{d}{\lambda(t,d)}\right)\left(1+\alpha\,\frac{\lambda(t,d)}{d}\right)\ , \qquad (6)$$

where

$$F\left(\frac{d}{\lambda(t,d)}\right)=\left[1+\exp\left(-\frac{d/\lambda(t,d)-\beta}{\gamma}\right)\right]^{-1} \qquad (7)$$

and

$$\beta=3.866\ ,\quad \gamma=0.3\times10^{-2}\ .$$

The coefficients β and γ were obtained as parameters, interpolating the data with a method of nonlinear least squares. The continuous curve represents the best fitting with 0.06 standard error.

IV. SURFACE CRITICAL TEMPERATURE $t_{H_{c3}}$

It is well known that surface superconductivity can be detected experimentally when $H_{c3} > H_{cF}$. Thus, recalling Eq. (1) we can observe H_{c3} for values of GL parameter $k(t, d)$ larger than a critical value $k_{H_{c3}}$ (at which $t = t_{H_{c3}}$) so that $H_{c3} = H_{cF}$. Equating (1) and (2), we obtain

$$k_{H_{c3}}=0.42\left(1+\alpha\,\frac{\lambda(t_{H_{c3}},d)}{d}\right)\ ; \qquad (8)$$

and substituting for $k(t, d)$,

$$k(t,d)=[2\pi\sqrt{2}\,H_c(t)\lambda^2(t,d)]/\Phi_0\ , \qquad (9)$$

we have the following equation for $t_{H_{c3}}$:

$$t_{H_{c3}}^6+(1-2a)t_{H_{c3}}^4+(a^2+b^2-1)t_{H_{c3}}^2+b^2-(a-1)^2=0\ , \qquad (10)$$

where

$$a=2\pi\sqrt{2}\,H_c(0)\lambda^2(0,d)/0.42\Phi_0$$

and

$$b=\alpha\lambda(0,d)/d\ .$$

Equation (10) yields only one real solution which allows us to predict the film thickness dependence of $t_{H_{c3}}$. The values of $t_{H_{c3}}$ and $k_{H_{c3}}$ calculated and measured for all the samples analyzed are summarized in Table I. The calculated values of $t_{H_{c3}}$ are derived from Eq. (10) using the parameters $\lambda(0,d)$ and α experimentally obtained by fitting the critical magnetic fields relative to each sample.

Finally, it can be observed that the experimental values of $t_{H_{c3}}$ are systematically smaller than the calculated ones.

A possible explanation for this situation is that the theoretical expression is derived by equating H_{cF} and H_{c3}, whereas it is possible to observe H_{c3} only if $H_{c3} > H_{cF}$.

V. CONCLUSIONS

Some interesting data have emerged from the experimental results.

(i) The order of the magnetic transition as a function of the thickness and the temperature has been studied. For films thinner than a critical thickness d_c, the transition is of the second order for all values of t, while for $d > d_c$ we determined a critical temperature t_{d_c} for each sample such that for $t < t_{d_c}$ a first-order transition is present, while for $t > t_{d_c}$ a second-order transition occurs at H_{cF}. This critical temperature was derived from different methods and the values obtained are in good agreement with each other.

(ii) The measured critical magnetic fields H_{cF} are in excellent agreement with the GL theory for thin films and within the limit $d \gg \xi$. We have determined a semiempirical functional expression which fits all the experimental data concerning H_{cF}. This expression, which is valid for any thickness, is obtained by means of a nonlinear combination of the two expressions valid within the limits $d < d_c$ and $d \gg \xi$.

(iii) Moreover, we have determined both experimentally and theoretically the temperature dependence of the critical temperature $t_{H_{c3}}$ at which surface superconductivity appears. The values obtained are in good agreement with the theoretical predictions. The possibility of studying in detail the surface superconductivity depends on our experimental technique; in fact, microwave measurements are strongly sensitive to the state of the material near the surface.

(iv) We also note that the values obtained for $\lambda(0, d)$ by the fitting of H_{c3}, are in agreement with those of other workers.[1,5]

Finally, we present two tables which summarize the values of $\lambda(0, d)$, $k_{H_{c3}}$, $t_{H_{c3}}$, α, and t_{d_c} which may be derived from our measurements, and we compare them with the theoretical predictions.

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