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Superconductivity in ultrathin Pb films deposited on silicon*

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Superconducting Pb films 11 to 200 Å thick have been deposited in ultrahigh vacuum on the clean (111) surface of crystalline silicon. Low-energy-electron diffraction indicated that the Pb preferentially grows with the Pb (111) plane parallel to the substrate surface. Measurements of normal-state film resistivity and of the superconducting transition temperature T_c were taken *in situ*. The dependence of T_c on film thickness was essentially identical to that previously measured for Pb films grown epitaxially on crystalline PbTe. This result is discussed in view of the dissimilarities between the two substrate materials.

I. INTRODUCTION

In this paper we report new data for the thickness dependence of ultrathin Pb films grown in ultrahigh vacuum on single-crystal silicon and measured *in situ*. These data indicate that the superconducting transition temperature T_c of these Pb films grown on Si behaves, as a function of thickness, in the same manner as for Pb films grown on the very different substrate PbTe.[1] On the basis of these data and earlier work done with Pb films deposited on polycrystalline SiO_x and Ge,[2] a model is developed in which a decreased superconducting interaction at the surfaces of crystallites of a microcrystalline film accounts for the T_c decrease in thinner films.

Experiments undertaken to understand the origin of changes in T_c of materials in thin-film form are numerous, and the effects involved may be lumped into several categories. Changes in the bulk properties of the material deposited, such as changes in lattice parameter or contamination of the film by gaseous impurities, will not be considered here, except to briefly mention that these effects cannot account for the present results. Changes in T_c due to the intrinsic nature of a thin film—the approach to a two-dimensional solid—are of the most fundamental interest. Included in such a category would be effects due to quantization of the electron wave function perpendicular to the plane of the film and the necessity to consider the boundary conditions at the film surface,[3] and substrate-induced changes in T_c.[4] Our experiments indicate that any changes in T_c induced by the nature of the substrate material are very small indeed, and that quantization or uniform-film-boundary effects cannot be separated from effects due to film granularity. The third category—effects due to the microstructure of the film—can also involve phenomena already mentioned, such as wave-function quantization and boundary effects, except that the grain or crystallite size of the film is the controlling parameter rather than thickness. Included in this category would be such phenomena as activated conduction between discrete grains, and changes in T_c due to changes in the electron-phonon coupling at grain or microcrystallite boundaries. This point will be discussed in more detail later on.

II. EXPERIMENTAL CONSIDERATIONS

The apparatus used in this experiment was essentially the same as used previously for the preparation, characterization, and *in situ* measurement of the T_c of Pb films deposited on PbTe,[1,5] and has been described elsewhere.[6] Modifications were made to the sample holder to accept the Si-crystal substrates, and a movable four-point resistive probe[7] was constructed to allow *in situ* measurement of Pb films deposited in succession on the electrically insulating Si.

As in previous experiments, an ultrahigh vacuum environment ($<10^{-9}$ Torr, 1.3×10^{-7} Pa) was maintained at all times to prevent substrate contamination or contamination of the film during and after evaporation. Low-energy-electron diffraction (LEED) and Auger-electron spectroscopy (AES) were used to monitor surface cleanliness and crystallinity, as discussed later. All measurements of T_c were made *in situ* to eliminate effects of exposure of the films to laboratory temperatures and pressures. In this way, oxidation of the ultrathin metal films and film agglomeration were eliminated.

The silicon substrates were cut from high-resistivity single-crystal silicon to expose a (111) plane, then polished mechanically and etched. The substrate was dumbbell shaped, as shown in Fig. 1, to allow the film end to be heated to near the melting point of Si for cleaning the surface. This was done by electron bombardment from a hot tungsten filament held at −2000 V. The other end was tightly clamped to the copper substrate

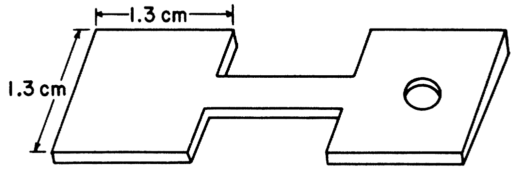

FIG. 1. Si substrate. The thickness was about 1 mm. The drilled end was clamped to an annealed Cu holder for good thermal contact.

holder for good thermal contact at low temperatures. Two Au-Fe versus chromel thermocouples were also tightly clamped to this end of the substrate, and a calibration made with thermometers cemented to the film end of the substrate after the film measurements were completed indicated that at low temperatures the error in reading film temperature was much smaller than the uncertainty of the individual thermocouple measurement (about ±0.1 K from near 2 to about 10 K). The thermocouples were calibrated at 7.2 K with a thick Pb film and at 3.7 K with a thick Sn film; temperatures in between were determined using a curve obtained by Rosenbaum.[8]

The appearance of a good silicon (111) 7 × 7 LEED pattern was used as the criterion for a suitable substrate surface. AES was used also to check surface cleanliness; a trace amount of carbon was the only contaminant detected on a cleaned surface.

The Pb films were deposited by evaporation from an electrically heated Ta oven source. The substrate temperature during evaporation was between 60 and 100 K. Each film warmed to approximately 100 K in the time (approximately $\frac{1}{2}$ h) between deposition and the start of cooldown, during which LEED and AES measurements were made. As long as the films were held below about 120 K, no differences were noted in crystallinity or coverage from run to run; above about 120 K the films appeared to agglomerate and became continuous at much larger thicknesses than the value of about 5–10 Å normally attained. Evaporation rates were about 50–60 Å/min; ambient pressure during evaporation was around 6×10^{-10} Torr (8×10^{-7} Pa).

The thickness of the Pb films was monitored by a quartz-crystal oscillator sensing head, which was calibrated with an interferometer at a film thickness of about 750 Å. Earlier work[5] with film resistivity and AES signals as a function of thickness indicated that deviations from linearity for the crystal monitor were not observable down to the smallest thicknesses. Sources of error in thickness measurements were primarily systematic due to a nonuniform deposition thickness across the substrate (± about 25%). This thickness variation depended somewhat on how full the source was and also on small variations in source-to-substrate geometry as the source was removed for refilling. The thickness variation across the region sampled by the measuring probes was much less, however, and was very constant from deposition to deposition in the same run. Therefore it was estimated that although the absolute error in thickness calibration from run to run might be as high as 10%, within one run the thickness error was determined only by oscillator drift, which gave an accuracy of approximately 0.5 Å or 5%, whichever was larger.

III. RESULTS

A LEED study of Pb deposited on Si (111) 7 × 7 has previously been reported by Estrup and Morrison, and epitaxial growth of Pb was observed at room temperature.[9] We also have observed epitaxy of Pb on Si (111) 7 × 7 at room temperature, but interpret our observations as resulting from the epitaxial growth of discrete islands of Pb, which gradually increase in size to cover the surface. Our LEED observations and resistance measurements indicated that at room temperature much more than 15 Å of Pb was needed to form a continuous metallic conducting film, whereas in Ref. 9, changes in conductivity were reported at room temperature with as little as one monolayer of Pb deposited. Unfortunately no cooling was available in the experiment reported in Ref. 9 so it is not clear whether those thinnest films exhibited metallic or activated conduction.

At lower temperatures, near 100 K, the Pb film grew differently. The LEED pattern background intensity increased much more rapidly with thickness, and by about 15 Å the Si 1 × 1 spots were completely eliminated. The spots from epitaxial Pb were much weaker and fuzzier than for Pb deposited on Si (111) at room temperature or for Pb deposited on PbTe (100) at 77 K.[5]

The conductivity of the silicon substrates at low temperatures was too low to measure (<10 μmho). With the substrate at room temperature, measurable changes in conductance occurred for as little as 5 Å of Pb, however this conductance decreased slowly upon cooling the substrate, indicating activated conduction. The thinnest superconducting films (~11 Å) deposited at about 80 K also showed some decrease in conductance with decreasing temperature down to about 6 K. Thicker films exhibited more metallic behavior, increasing slightly in conductance as the temperature was lowered. The conductivity of these films at about 10 K increased approximately linearly with thick-

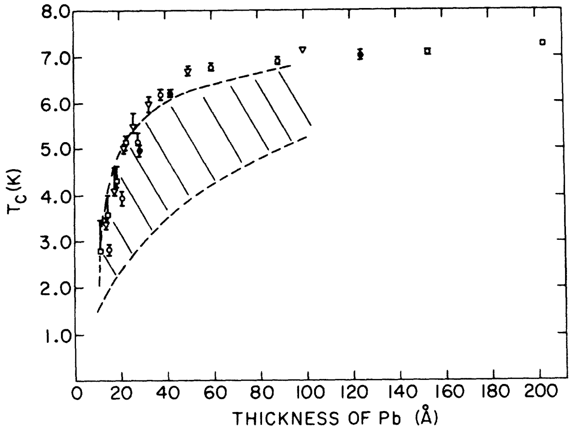

FIG. 2. T_c as a function of thickness (in angstroms) for Pb deposited on Si (111), ●, ○, □; Pb deposited on PbTe (100), ▽ (from Ref. 1); Pb deposited on evaporated Ge or SiO_x, shaded area (from Ref. 2).

ness, as discussed later. The sheet resistance of the thinner films approached 3000 Ω/square, much greater than the earlier Pb films deposited on PbTe,[5] but similar to values reported for Pb films deposited or SiO_x and Ge.[2] The behavior of film conductivity as a function of thickness will be discussed in more detail later.

The behavior of the film T_c as a function of thickness is shown in Fig. 2, along with data for Pb on PbTe from Ref. 5 and Pb on SiO_x and Ge from Ref. 2. Within the accuracy of the data, T_c for Pb on Si decreases with decreasing film thickness identically to T_c for Pb on PbTe. These data also approximately form an upper bound on the range of values from Ref. 2. A model to explain this behavior is discussed in Sec. IV.

IV. MODEL OF SUPERCONDUCTING FILM

The model proposed here is not intended to represent a detailed microscopic description of the superconducting films of this experiment. However, by using the concept of a microcrystalline film with a reduced pairing interaction at the crystallite surfaces, the behavior of film resistance and superconducting T_c as a function of thickness can be described. The similarity between data of Pb on Si and Pb on PbTe leads to conclusions about the magnitude of the film-substrate interaction. This model is very similar to models previously proposed,[2,10,11] and connections with these and other descriptions of thin film superconductivity will be discussed.

The data to be discussed here include the results of this experiment and also of previous investigations where Pb was deposited on PbTe,[1,5] and on SiO and Ge.[2] If we consider a model in which boundaries between microcrystallites of the film contribute the greatest part of the low-temperature normal-state resistance, then the large difference between the sheet resistance of films deposited on PbTe (~2 Ω/square) and on Si (~3000 Ω/square) can be attributed to the conducting PbTe substrate shorting out the high-resistance grain boundaries of the film. If the model also allows the crystallites to grow in (average) size as the film becomes thicker and more grains coalesce into larger crystallites, the linear increase of conductivity of Pb films on Si can be obtained from a simple model of grain behavior.

In this model, the film is considered to consist of grains or crystallites which extend from the substrate to the film surface, across the thickness of the film. The grain boundaries are perpendicular to the substrate and contribute to the film resistivity by acting as electron scatterers. The surface and film-substrate interface also will contribute to the film resistivity due to nonspecular reflection of electrons from these boundaries.[12] Superconductivity may be suppressed due to a reduced superconducting interaction at the film-substrate or film-vacuum boundaries, and also at grain boundaries.

A. Film resistance

A model such as is described above has been used by Mayadas and Shatzke to calculate the resistivity of thin films under conditions where bulk scattering, surface scattering, and grain boundary scattering all contribute to the measured resistivity.[13] In their model, Mayadas and Shatzke use δ-function potentials parametrized by a reflection coefficient R to simulate the effect of grain boundary scattering. Scattering from the film surfaces is parametrized by the specularity of reflections P. The grain boundary positions are located according to a Gaussian distribution with an average separation (grain size) d. Mayadas and Shatzke obtain, for the conductivity of the film,

$$\sigma_F = \sigma_g - \sigma_S \tag{1}$$

and

$$\sigma_F = \sigma_g - \frac{6\sigma_0}{\pi K_0}(1 - P) \times \int_0^{\pi/2} d\phi \int_1^\infty dx \frac{\cos^2\phi}{H^2(x, \phi)} \left(\frac{1}{x^3} - \frac{1}{x^5}\right) \times \frac{1 - e^{-K_0 t H(x, \phi)}}{1 - P e^{-K_0 t H(x, \phi)}}, \tag{2}$$

where σ_g, the conductance considering grain boundary scattering alone, is given by

$$\sigma_g = 3\sigma_0[\tfrac{1}{3} - \tfrac{1}{2}\alpha + \alpha^2 - \alpha^3 \ln(1+1/\alpha)] \tag{3}$$

and

$$H(x,\phi) = 1 + \alpha[\cos\phi(1 - 1/x^2)^{1/2}]^{-1}. \tag{4}$$

The thickness-dependent parameter is $K_0 = t/l_0$, and also $\alpha = (l_0/d)[R/(1-R)]$. Equations (2) and (3) are Eqs. (15a) and (10) from Ref.13. The conductivity which an infinitely thick single crystal would have is σ_0, t is the film thickness, and l_0 is the single-crystal mean free path. Under the condition that the grain size d is proportional to the thickness $d = t/\beta$, we have $\alpha = A/K_0$, where

$$A = \beta R/(1-R). \tag{5}$$

Therefore the only parameters used in this description of film conductivity are A, which is determined by the scattering strength of the grain boundaries and by the ratio of grain size to film thickness, and P, the specularity of reflections from the film surface.

Values of σ_F from Eq. (2) must be computed numerically. It can be seen that for very thin films, which give small values of K_0, the double integral will not depend very strongly on P. This is because, for small film thicknesses and thus small grain sizes, the effective mean free path is greatly reduced, limiting the effect of surface scattering on the resistivity. In this limit the electron mean free path will be completely determined by the grain size, and the bulk mean free path l_0 cannot enter the expression for σ_F.

A plot of $\rho_F/\rho_0 \equiv (\sigma_F/\sigma_0)^{-1}$ as a function of K_0 is shown in Fig. 3, for $A = 2.0$, $P = 0.5$, and $l_0 = 2.8 \times 10^4$ Å. The value for ρ_0 was taken to be 5.4×10^{-8} Ω cm at 10 K,[14] and l_0 was determined from this by the empirical relation $\rho_0 l_0 \cong 1.5 \times 10^{-11}$ Ω cm^2.[15] This is compared in Fig. 3 to the unadjusted values of the film normal-state resistivity ratio ρ_F/ρ_0 taken near 10 K during one of the runs of Pb on Si, and using the same value of ρ_0 used to determine l_0 above. The agreement is good enough to conclude that this type of model may have some validity even for such ultrathin films. The slope of the line is very close to −1.0, indicating that $\rho_F/\rho_0 \propto K_0^{-1}$. Since $\rho_0 \propto l_0^{-1}$ and $K_0 \propto l_0^{-1}$, ρ_F has no dependence on l_0, as the case should be. Indeed, $\rho_F \propto t^{-1}$, and the thickness dependence of resistivity is just what is to be expected from a film in which grain size, and thus effective mean free path, decreases linearly with thickness.

In assessing the applicability of this type of model to ultrathin films, it should be kept in mind, however, that for the thinnest films ($t \cong 11$ Å) effects due to wave-function quantization may become significant, which has not been accounted for in the model. Also, the use of $A = 2.0$ implies, with grain size equal to thickness, that $R \cong 0.67$. Such a large reflectivity may indicate a bumpy or partially discontinuous film.

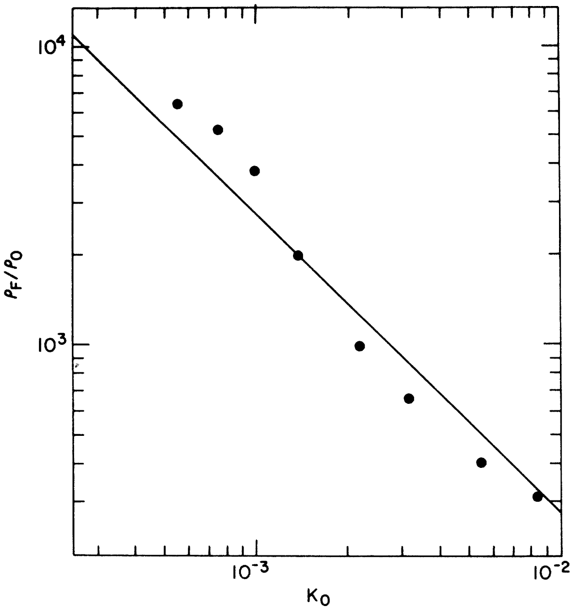

FIG. 3. Film resistivity ratio ρ_F/ρ_0 as a function of K_0, calculated from Eq. (15a) of Ref. 13 [Eq. (2) of this text]. ●, data for Pb deposited on Si. Parameters used in the calculation of ρ_F were $A = 2.0$, $P = 0.5$, $l_0 = 2.8 \times 10^4$ Å.

For Pb films deposited on PbTe, the measured thickness-dependent film resistivity for the thinnest films is several orders of magnitude lower than that of films deposited on silicon, and does not show as large a dependence on thickness as it does in the case of Si substrates. This can be understood in terms of the shorting of the highly resistive grain boundaries by the conducting PbTe substrate. As the film thickness and average grain size increases, both the resistance of a grain and the resistance due to (shorted) grain boundaries will decrease approximately as t^{-1}. For a given thickness, the relative contribution to the film resistance from grain resistance or shorted grain boundary resistance will depend on the relative magnitudes of the film and substrate resistivity and the magnitude of the film-substrate contact resistance. In Fig. 4, film conductance, $\sigma_\square$, is plotted as a function of thickness for a Pb film deposited at about 80 K on a PbTe substrate. The slope of a straight line drawn through the points indicates an effective film resistivity of about 2.7×10^{-6} Ω cm. Since the bulk resistivity of Pb is about 7×10^{-6} Ω cm,[14] at 100 K, the temperature at which the measurements on PbTe were taken, it appears that the grain boundaries in this

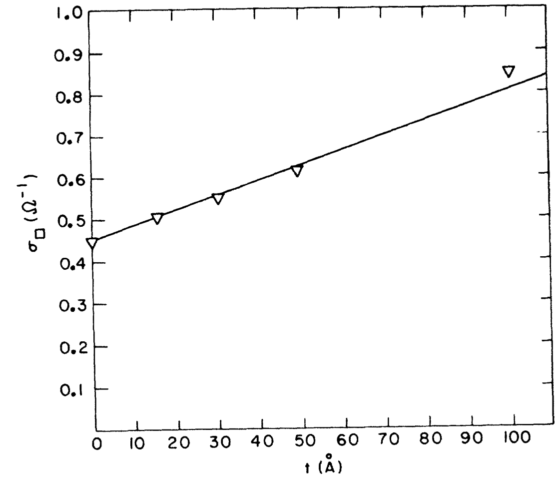

FIG. 4. Film conductance as a function of thickness, for Pb deposited on PbTe. The slope of the straight line indicates an effective film resistivity of 2.7×10^{-6} Ω cm.

case are well shorted by the substrate. This in turn implies that any film-substrate contact resistance is small compared to grain boundary resistances.

B. Film superconductivity

In an earlier paper we analyzed the decrease in T_c with a decrease in film thickness in terms of a Cooper-limit proximity-effect model.[1] This model has also been used by a number of other researchers[2,10,11] and leads to a T_c dependence

$$\ln(T_{c0}/T_c) = N_n d_n / N_s^2 V_s d_s\,, \tag{6}$$

where T_{c0} is the bulk T_c, N_n (N_s) the normal (superconducting) material density of states at the Fermi level, V_s is the superconducting interaction parameter, and d_n (d_s) is the normal (superconducting) material thickness. Essentially this model, valid only for very thin layers, averages the coupling NV by the amount of time an electron spends in each layer. The weighting factor will be the density of states multiplied by the layer thickness. For a uniform film in which the normal layer is external to the film, $d_n = \text{const}$, and $d_s = t$, so that

$$\ln(T_{c0}/T_c) = N_n d_n / N_s^2 V_s t\,. \tag{7}$$

For a film in which the normal material, or material with reduced NV, resides in grain boundaries and surfaces of grains as in our model which was used to describe film resistivity, the ratio R_{ns} of normal to superconducting material is just the ratio of surface to volume for adjoining grains. If we take, for example, square grains with width proportional to thickness, $d = t/\beta$;

$$R_{ns} = \frac{2d_n t^2/\beta^2 + 2d_n t^2/\beta}{t^3/\beta^2} \tag{8}$$

if the "normal" layer separating the crystallites and which also exists on the substrate and vacuum interfaces is much thinner than the film thickness. From this we obtain

$$\ln(T_{c0}/T_c) = (N_n/N_s^2 V_s)(2d_n/t)(1+\beta)\,. \tag{9}$$

Again the same t^{-1} thickness dependence holds as was obtained for a uniform film without grain boundaries.

In Fig. 5, $\ln(T_{c0}/T_c)$ is plotted as a function of t^{-1} for data from this experiment and the two previous experiments for Pb on PbTe and Pb on SiO_x and Ge. The fit to a straight line is not particularly good: both the Pb films on crystalline substrates and the upper-T_c boundary of the films deposited on SiO_x and Ge show a greater decrease in T_c for the thinnest films than is predicted by the proximity-effect model. However, the general trends of the data are represented by this type of calculation. It should be mentioned that both a microcrystalline film as modeled above and a uniform single-crystal film would exhibit the same linear behavior on this type of plot, with different slopes, so that the location of any normal material contributing to a proximity effect cannot be determined from this sort of analysis.

The curvature in the proximity-effect plot can be explained by an extension of our model to include an irregular or bumpy film surface, which grad-

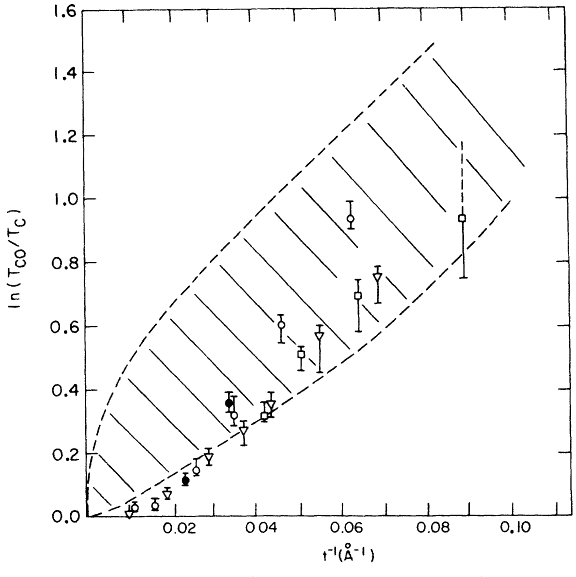

FIG. 5. Plot of $\ln(T_{c0}/T_c)$ as a function of t^{-1}, for Pb deposited on Si (111), ●, ○, □; Pb deposited on PbTe, ▽ (from Ref. 1); Pb deposited on evaporated Ge or SiO_x, shaded area (from Ref. 2).

ually smooths out as the film becomes thicker. This can introduce a term into Eq. (9) for R_{ns} which goes as t^{-2}, and gives an upward curvature to the plot. This film-roughness or film-granularity term has been discussed by Yu *et al.*[3] Another possible cause for a T_c which is decreased below that predicted by the proximity model would be fluctuation effects, which could possibly reduce T_c in the thinnest films by as much as 1 K. This has been discussed by Strongin *et al.*[2]

One conclusion can be drawn from this data without reservation, and that is that the choice of substrate material *per se* has little or no effect on the T_c behavior of these Pb films. This suggests that, contrary to our expectations, little or no proximity effect arises from the substrate material itself, unless by some amazing coincidence the PbTe and Si substrates, and also those SiO_x and Ge substrates which gave the highest-T_c Pb films, all had nearly the same density of states at the interface. The implication of this surprising finding is also discussed at more length in Sec. V.

V. DISCUSSION

The model we have presented, in which growing crystallites in a microcrystalline film account for the resistance behavior in terms of grain boundaries, and a proximity effect approximates the T_c behavior, adequately accounts for the observed behavior of Pb on PbTe and Pb on Si. If we consider the scatter in the data for Pb on SiO_x and Ge to be due to variations in granularity caused by variations in deposition parameters, we can identify the higher-T_c films with less granular, more crystalline growth, with less surface area. Thus since the high-T_c boundary of this earlier data is very close to the data for Pb on PbTe and on Si, our model also qualitatively explains the Pb on SiO_x and Ge data.

Despite the general applicability of our model as a guide to understanding some of the processes affecting T_c in these films, there are several points remaining to be explained. First, the curvature in the proximity-effect plot could have several origins. If the film surface is wrinkled or if the film contains holes, which raises the surface-to-volume ratio of the thinner films, T_c will be depressed more rapidly for the thinner films than is predicted from the proximity model we have considered. Also, if the "normal" layer is actually a portion of the film itself and is a layer of constant thickness, at very small thicknesses the volume occupied by the normal layer becomes an appreciable fraction of the total film volume. This too would lead to a curvature in the proximity plot in the direction observed, but primarily only at very small film thicknesses. Finally, as also mentioned previously, we should consider the possibility of fluctuations lowering the T_c. This effect will generally increase for films with higher $R_\square$, as discussed in Ref. 2.

The physical origin of the proximity-effect lowering of T_c at this stage can only be guessed at. The normal material of the Cooper-limit model could be any material with a lowered pairing interaction. The lowering of T_c to be expected from a change in phonon spectrum which might occur at crystallite surfaces is small in Pb,[16] so that if the film itself had a dead region one would expect the electronic properties to be altered in that retion. Electron wave-function leakage past the ion core boundary of the film is a possible cause of T_c lowering,[3] but if this is the case, the lack of measurable difference between T_c behavior for films deposited on the small gap (about 0.2 eV) electrically conducting PbTe and films deposited on the larger gap (1.2 eV) electrically insulating Si is hard to understand. Also, calculations have been made by Yu *et al.*,[3] in which quantization of the electron-wave-function perpendicular to the film surface is taken into account and in which careful consideration is given to appropriate boundary conditions. They obtain curves in which the T_c of the thinner films is depressed *less* than is obtained from the Cooper-limit calculation, and find it necessary to introduce a surface roughness term to describe experimental results. The concept of T_c lowering by pair-weakening due to the scattering of conduction electrons into localized states at the surface, grain boundaries, or the film-substrate interface should also be mentioned.[17] This has the attractive feature of explaining the lack of differentiation of behavior for films on PbTe and Si, since presumably the localized states could be a consequence of disorder at the crystallite surfaces and interfaces, and need not involve the substrate material directly at all.

A further question is raised by our model. Since the film resistance and T_c behavior depend on the geometry of the crystallite growth, we are forced by the superconductivity data to conclude that either crystallite growth for PbTe and Si substrates are very similar, or that the boundaries *between* grains have little effect on T_c. Identical crystallite growth would be quite a coincidence, given the different symmetry of PbTe (100) and Si (111), although LEED shows Pb cystallites growing in both cases with the same (111) face parallel to the substrate surface. These points are not clear at this time.

It is tempting to generalize this model to explain data from other experiments. Several other authors have explained their results via a proximity-effect model. Grandquist and Claesson[10] have used the concept of a "dead layer" at the surfaces of their amorphous Bi and Ga films to explain the origin of the proximity effect lowering of T_c. Strongin *et al.*[2] also used the concept of a surface layer with a decreased interaction to describe their Pb films on SiO_x and Ge, as did Wolff *et al.*[11] to describe their ultrathin Nb films. Variations on this approach have been used to describe T_c changes in other materials. For example, Pettit and Silcox[18] have correlated T_c increases in granular Al films with inverse grain size, invoking an enhanced interaction at the grain surface due to a modified phonon spectrum.

In all these cases the enhancement or decrease in T_c depends on the surface of the film. As a consequence, the granularity, and thus surface area, of the film is the controlling parameter rather than thickness. Thus future experiments must be carefully conducted to closely control such parameters, which apparently are still of importance even in such carefully grown epitaxial films as reported here and in Refs. 5 and 1. Additionally, the details of the surface interaction are not clear, since in this experiment no measurable difference in T_c was detected between films deposited on PbTe and on Si, yet several experiments have shown that overcoating thin superconducting films with dielectric materials can have fairly large effects on T_c.[2,19-21]

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