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Evidence for Berezinskii–Kosterlitz–Thouless transition in atomically flat two-dimensional Pb superconducting films

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ABSTRACT

We report results from scanning tunneling microscopy and transport measurements on a series of crystalline lead films containing an integer number of atomic layers, and find that the observed features in sufficiently thin films are consistent with Berezinskii–Kosterlitz–Thouless (BKT) physics. Specifically, Cooper pairing and superconductivity disappear at two distinct temperatures; the current–voltage characteristics in the intermediate phase are non-Ohmic; and the temperature and current dependences of resistance agree with the expectation from the BKT theory.

1. Introduction

Superfluid phase transition behaves very differently in three and two dimensions. Both are described by a macroscopic complex wave function $\psi_0(r)e^{i\varphi(r)}$, thought of as the order parameter, with two degrees of freedom: the amplitude ψ_0 and the phase φ. In three dimensions superfluidity is destroyed when the amplitude of the order parameter vanishes. For two dimensional (2D) systems, the Berezinskii–Kosterlitz–Thouless (BKT) theory predicts that superfluidity vanishes when the phase correlations change from an algebraic long range order to an exponential decay, caused by the physics of vortex unbinding at a critical temperature T_φ [1,2]. Experimental realization of the BKT model was established in superfluid helium films more than 30 years ago [3,4]. Whether the BKT physics applies also to quasi-2D superconductors has been a question of considerable interest. Strictly speaking, the BKT model is not applicable to infinitely large thin superconducting films because the interaction between the Pearl vortices [5] falls off as $1/r$ rather than $\ln(r)$ at long distances, which implies that isolated vortices have a finite energy and can be thermally excited at all temperatures. In a pioneering work, however, Beasley et al. [6] argued that when the perpendicular penetration depth of the film, $\lambda_\perp = \lambda^2/d$, where λ is the penetration depth of the three dimensional (3D) system and d is the film thickness, is larger than the sample size, the interaction between the Pearl vortices is of the form $\ln(r)$ over the entire sample, thus opening the possibility of BKT transition in superconducting thin films as well.

A number of experiments on amorphous or granular films and arrays of Josephson junctions exhibit signatures that are consistent with BKT mechanism [7–11]. Nonetheless, questions have been raised regarding the interpretation of the experiments and the applicability of various theoretical results to the regime where experiments are often carried out [12–15]. In particular, it has been argued that the "fugacity" of the vortices is so large as to produce a sizable density of vortex–antivortex pairs near the transition temperature, and the BKT theory, which assumes a dilute limit, cannot be applied in its standard form; it has been suggested that a large density of vortex–antivortex pairs may even cause them to crystallize [12]. In this work, by studying crystalline and atomically flat films of Pb containing an integer number of atomic layers grown on Si(111) by molecular beam epitaxy (MBE) [16–20], we are able to investigate systematically the nature of superconductivity in 2D films with a combination of scanning tunneling microscopy/spectroscopy (STM/STS) and transport measurements.

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2. Materials and methods

The molecular beam epitaxy (MBE) growth and scanning tunneling microscopy (STM) measurements were carried out in a commercial ultra-high vacuum MBE–STM system (Omicron) with a base pressure of 1.5×10^{-10} Torr. The atomically flat ultrathin crystalline Pb films over a macroscopic area of 3×5 mm^2 were grown on a Pb-induced striped incommensurate (SIC) 'wetting' layer on Si (111) [20]. The thickness range of the films studied is from 3 to 51 monolayers (MLs). Our definition of the number MLs includes the 'wetting' layer; this convention is different from that used in other papers reporting STS study in Ref. [18,19]. A typical STM image of the atomically flat 3 ML films is shown in Fig. 1A. The steps seen in Fig. 1A originate from the Si substrate; the film consists of exactly the same number (3 ML) of atomic Pb layers on all the terraces. Thicker films exhibit the same morphology.

For *ex situ* transport measurement, amorphous Si capping layers were deposited on the Pb films at 100 K. The Si capping layer does not damage or degrade the crystalline order of the Pb films (Fig. S1A). Fig. 1B shows the surface morphology of the completed Si capping layer with a nominal thickness of 20 nm. The Si capping layer appears to be very effective in preventing oxidation of the Pb films during the short period (~60 min) when the samples are in ambient environment being prepared for transport measurements. The schematic for four-probe transport measurement is shown in Fig. S1B. The contact resistance between the In electrodes and the films are found to range reliably between 60 and 150 Ω. Since the Si substrate used is insulating below 30 K the Si substrate and the amorphous Si capping layer form an ideal platform for transport measurements of the Pb films.

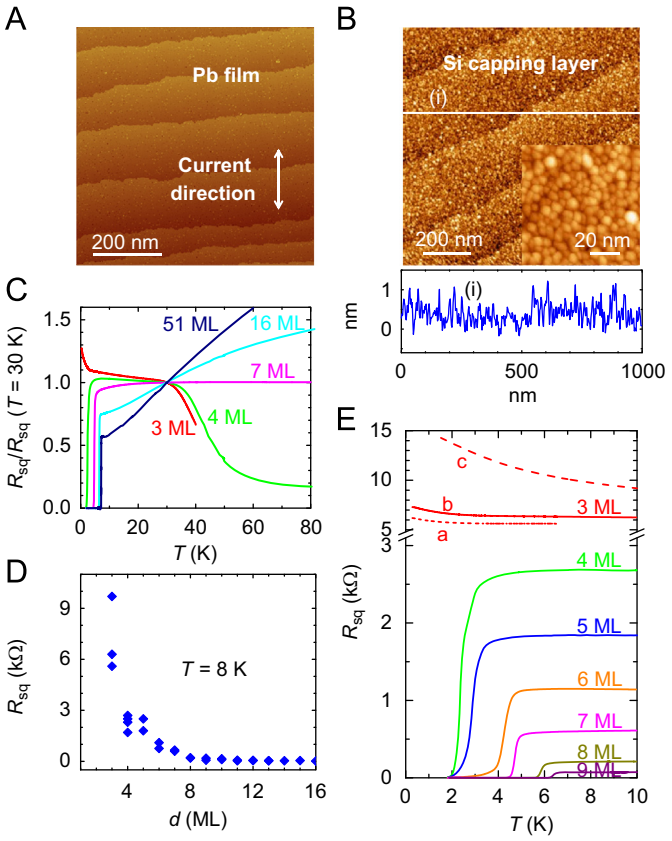

Fig. 1. STM images, the temperature and thickness dependences of the sheet resistance R_{sq} of crystalline Pb films. (A) A typical STM image of the 3 ML Pb film [2 ML crystalline Pb film grown on a Pb-induced SIC wetting layer on Si(111)]. The terrace width of Si(111) substrate is 100–300 nm. All crystalline films studied here exhibit the same morphology. (B) An STM image after deposition of a 20 nm thick Si capping layer. The inset is a zoomed-in image. The roughness of the film (~1 nm) is shown below the STM image. (C) $R_{sq}(T)$ (normalized at 30 K) for several films. R_{sq} at $T=30$ K are 5850, 2590, 634, 26.5 and 9.2 Ω, respectively. (D) Normal-state resistance R_{sq} at $T=8$ K as a function of d. Substantial d-dependence is found for $d=3$–6 MLs. The superconducting transition temperature,T_φ , for the films is governed primarily by d and not the normal-state resistance. (E) Evolution of$R_{sq}(T)$ from 0 to 10 K for the films of 3 to 9 ML. The 3 ML film remains insulating down to 0.29 K. The excitation current is 1 μA.

3. Results and discussion

The sheet resistances $R_{sq}(T)$ were measured as a function of temperature for a total of 34 samples with thickness d between 3 and 51 ML. The resistances for some of the samples, normalized at 30 K, are shown from 0 to 80 K in Fig. 1C. The results between 8 and 30 K nicely illustrate the evolution from insulating to metallic behavior as d is increased from 3 to 51 ML. While the 16 and 51 ML films show standard metallic behavior (The 51 ML film has a superconducting transition temperature of 7.2 K, identical to that of bulk) and the 3 ML films were found to be insulating, the behavior of the 4 and 7 ML films straddles the boundary between the insulating and metallic states prior to the onset of superconductivity. The normal state R_{sq} (measured at 8 K) of a number of films are shown in Fig. 1D. The temperature dependence of R_{sq} for the films from 3 to 9 ML at low temperature is shown in more detail in Fig. 1E. The 3 ML films show insulating behavior down to 0.29 K. For samples with $d>3$, a clear superconducting transition can be seen. The transition temperature T_φ can be defined either as the temperature where the resistance falls to half its normal state value (Fig. S2), or from a more detailed fitting procedure (Fig. S3), and is plotted in Fig. 2 as a function of d. STS studies were also carried out on films of 3–7 ML. The tunneling spectra show a superconducting gap Δ below a thickness dependent Bardeen–Cooper–Schrieffer (BCS) pairing temperature T_Δ (Fig. S4). T_Δ of the 3–7 ML films are displayed in Fig. 2. Two key results of this experiment are that T_φ's for films of $d=4$–9 ML scale linearly with d and that T_Δ's for films of $d=4$–7 ML are distinctly higher than T_φ's. T_Δ increases rapidly from 1.8 K for the 1 ML film

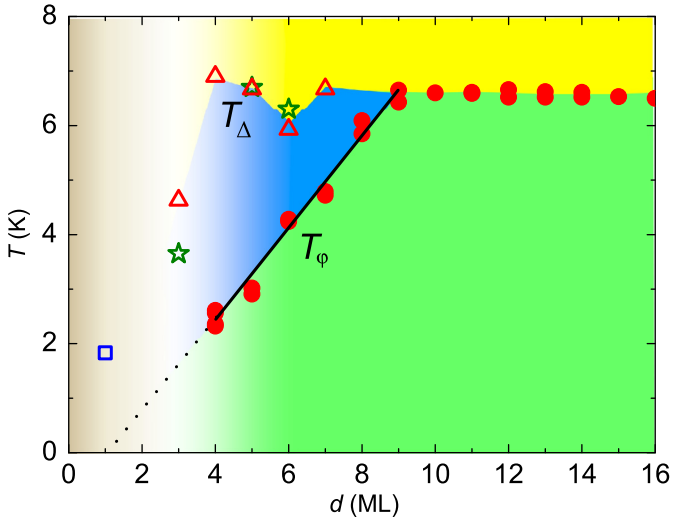

Fig. 2. (Color online) Phase diagram of the 2D Pb films displaying superconducting (Green), phase fluctuating (Blue), metallic (Yellow), and insulating (Gray) regions. Films of $d\leq3$ ML are driven insulating by disorder. The solid circles show T_φ, as determined by transport measurements. The black straight line is a linear fit of the data for films with d between 4 and 9 ML. It extrapolates to $d=1$ ML at $T=0$. The open symbols show the BCS pairing transition temperature T_Δ; red triangles are from this experiment (see SI text) whereas green stars and blue square are taken from previous experiments in Ref. [18, 19]. T_Δ's for films with $d=4$–7 ML are distinctly higher than T_φ's.

[20] to 6.9 K for the 4 ML film (Fig. S4), and then exhibits an oscillatory thickness dependence due to quantum confinement of finite thickness [16,18,19]. T_Δ's for films with d≥8 ML on the SIC wetting layer were not measured but an experiment [18] on films with d between 6 and 16 ML on un-reconstructed wetting layer found T_Δ to vary less than 0.1 K for d≥7 ML. This suggests the T_Δ for our films of $d>7$ ML are close to that of the 7 ML film or 6.7 K.

We note that, for small d, T_φ and T_Δ have distinct phase boundaries, in contrast to thicker films where the two transitions occur simultaneously. For the 4–7 ML Pb films, for a range of temperatures (shaded in blue in Fig. 2) Cooper pairs exist but do not superconduct. This behavior is distinct from that observed in typical 3D superconductors, where the bottleneck is Cooper pair formation, not phase coherence; as soon as Cooper pairs are formed, they Bose condense.

It is most natural to ascribe the behavior in 4–9 ML films to the BKT phase transition in 2D. The transition implies that for very small T, the amplitude of the macroscopic wave function is nonzero and its phase has quasi long range order, producing a zero resistance. At T_φ, the algebraic phase coherence is destroyed by free and mobile individual vortices, although the superconducting gap (or the amplitude of the order parameter) measured by STS remains nonzero. Eventually, when the temperature is raised further, at T_Δ Cooper pairs vanish and the film becomes normal. In what follows, we provide strong evidence that the superconducting phase transition at T_φ is of the BKT type.

We begin with the temperature dependence of the resistance. It was predicted that if the superconducting transition of a 2D film is governed by the BKT physics, the sheet resistance R_{sq} near T_φ will show a temperature dependence of the form [21]

$$R_{sq}(T) = R_0 \exp[-b(T/T_\varphi - 1)^{-1/2}], \quad (1)$$

where R_0 and b are sample specific fitting parameters. The value of T_φ can be readily determined from the derivative of R_{sq} as a function of T. We have carried out such analyses for all films studied (Fig. S3) and find that Eq. (1) provides a good description of $R_{sq}$and yields sensible transition temperatures for films with d less than 16 ML. For thicker films, R_{sq} drops abruptly at the superconducting transition temperature, resembling that of a bulk superconductor. For films with d≥9 ML, the value of T_φ as deduced via Eq. (1) saturates abruptly at 6.6 ± 0.15 K, identical to T_Δ of the films, indicating that these films are in the 3D limit and the transition is BCS in nature. If we define $T_{1/2}$ as the temperature where R_{sq} is reduced to half of the normal state value, then the values of $T_{1/2}$ are found to be within a few mK of T_φ. And thus, using $T_{1/2}$ instead of T_φ as the superconducting transition temperature does not make any material difference to the phase boundary (Fig. S2).

We note that Eq. (1) is valid at temperatures close to T_φ but sufficiently below the fluctuation corrected mean field temperature T_{c0}. We have used the relation from Aslamasov–Larkin theory $R_{sq}^{-1} = R_N^{-1} + R_0/(T/T_{c0}-1)$ which relates the sheet resistance R_{sq} to the normal sheet resistance R_N, to estimate T_{c0}. For 4 (5) ML samples we find that T_{c0} is ~2 (1) K above the estimated T_φ, and the two temperatures merge, as expected, as the film thickness is increased. It is also noted that the temperature dependence of the resistance in the tail is not described by the BKT theory; this has been noticed before by Hsu and Kapitulnik in Nb films [22] and by Reyren et al. in oxide layers [23] and attributed [22] to either a residual magnetic field or to disorder that pins unbound vortex antivortex pairs.

Strong support for the BKT physics is found in the voltage–current (V–I) isotherms measured over a fine grid of different temperatures for the 4 ML film. The evolution of these isotherms when the temperature is varied across T_φ is shown in Fig. 3A, and is precisely what is expected for a system governed by the BKT mechanism. A small but finite resistance is found at temperatures below T_φ. This is the case because in the BKT scenario, zero resistance is possible only in the zero current limit; any small non-zero current will exert a force of opposite signs on vortices of opposite polarity, breaking apart the most loosely bound vortex–anti-vortex pairs and thereby giving rise to finite resistance and voltage. Because the fraction of the dissociated vortex pairs depends on the current, a non-ohmic behavior is found. At even higher currents, after most of the pairs are broken apart, the rate of increase of V with I is reduced and the V–I curves revert toward ohmic. In the region where the voltage shows the most rapid increase, it varies as $I^{\alpha(T)}$ with a temperature dependent exponent $\alpha(T)$. The exponent $\alpha(T)$ is greater than 3 below T_φ equal to 3 at T_φ and between unity and 3 in the range $T_\varphi < T < T_\Delta$ [9–11,21,24]. The V–I curves conform to this behavior and α is found to be 3 at 2.46 K. For comparison, T_φ deduced via Eq. (1) from the $R_{sq}(T)$ curve at low excitation current for this sample is 2.61 K (Fig. S3A). We attribute the slight discrepancy to heating effects. Since the $V=I^3$ dependence is found at a substantial current, it is probable that the I^2R heating creates a temperature gradient between the sample and the temperature sensor position. As a result, the sensor underestimates the temperature, which might account for the discrepancy.

While not obvious from Fig. 3A, the V–I curves measured at 4, 5 and even 6 K are sub-ohmic. This behavior is revealed clearly in Fig. 3B and C which display the deviations in the voltage and the resistance from their values at 7 K. The evolution towards ohmic

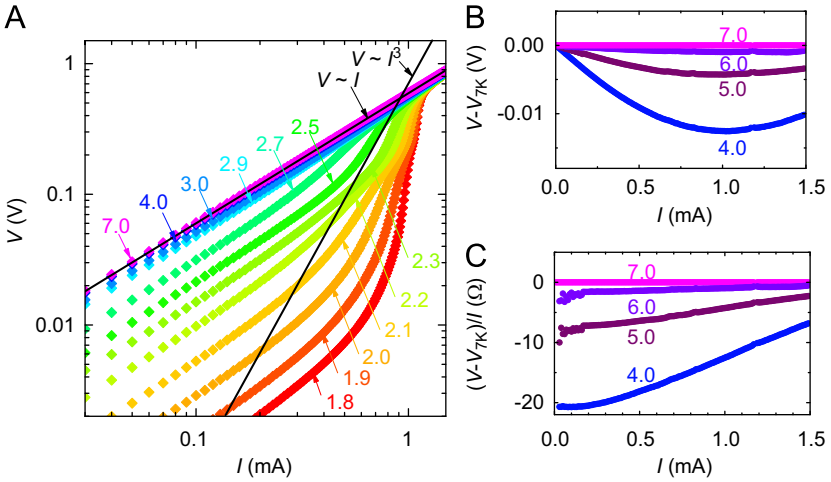

Fig. 3. V–I isotherms for the 4 ML film. (A) V–I curves at different temperatures (from 1.8 up to 7.0 K) on a log–log scale. The exponent $\alpha(T)$ defined by $V \sim I^\alpha$ decreases with increasing temperature and assumes the value $\alpha=3$ at 2.46 K, which is identified as T_φ. (B) V–$V_{7\,K}$ vs I plot for 4, 5, 6 K. (C) $(V$–$V_{7\,K})/I$ vs I plot. The resistance shows an evolution towards ohmic behavior with increasing current and as T is raised towards 7 K, the pairing temperature T_Δ of the 4 ML film.

behavior with increasing current and as temperature is raised towards T_Δ (i. e. ~7 K) is consistent with the physics of the destruction of superconductivity due to unbound vortices.

A linear extrapolation of the T_φ line in Fig. 2 would predict a T_φ of 1.7 K for the 3 ML film. However, as shown in Fig. 1E, all three 3 ML samples show insulating behavior down to the lowest temperatures in our study. STS measurements, on the other hand, find a superconducting gap that persists up to a temperature T_Δ of 4.63 K. The presence of nonzero amplitude for the order parameter up to 4.63 K in the 3 ML film is supported by the comparison between magnetoresistance (MR) measurements of the 3 ML and 4 ML films (Fig. S5). The temperature evolution of the MR curves of the 3 ML film from the lowest temperature (0.29 K) is similar to that of the MR curves of the 4 ML at temperature above T_φ. The MR effect of both films fades out near T_Δ. We believe that the physics of the qualitatively different behavior of the 3 ML films is the same as that of superconductor to insulator transition studied previously in quench condensed films, which found that the low T phase is determined by whether the normal state sheet resistance is higher or lower than a critical value that is of order 6.45 kΩ [25]. The 3 ML films are 'intrinsically' more disordered because it is difficult to grow a 3 ML film with crystallinity comparable to that in the thicker films. The R_{sq} of the two 3 ML films (samples a and b shown in Fig. 1E) are indeed 5.8 and 6.7 kΩ at 1.7 K, the expected T_φ, close to the quantum resistance value. It would be interesting to examine if better quality 3 ML films will show a superconducting transition at 1.7 K.

We note that while the transport measurements have been performed *ex situ* on capped films, the STM measurements have been performed *in situ* on uncapped films. It is very likely that the additional disorder due to the capping of the layer is responsible for suppressing the resistive transition temperature T_φ. One may ask if the same disorder also suppresses T_Δ, the Cooper pairing transition. While *in situ* transport measurements are not possible with our current apparatus, the transport experiments on the capped films can also provide an estimation of T_Δ defined as the temperature below which *V–I* characteristics are nonlinear, as expected from the BKT theory. We find that (i) the *V–I* characteristics show nonlinearity at temperatures above T_φ, and (ii) nonlinearity persists up to temperatures that correlate with the pairing temperature T_Δ of the uncapped layers. These observations give us confidence that pairing transition temperature T_Δ is not significantly affected by silicon capping.

The linear dependence of T_φ on d is reminiscent to that found in superfluid helium films [3,4,26], where torsional oscillator measurements confirmed the BKT prediction of a universal jump in the superfluid density, n_s, that scales with T_φ and other constants when the temperature is cooled across T_φ [27]

$$k_B T_\varphi = h^2 n_s^{2D}(T_\varphi)/8\pi m k_B, \tag{2}$$

where m is the mass of ^{4}He atom and $n_s^{2D}(T_\varphi)$ is the 2D superfluid density of ^{4}He atoms just below T_φ. The same expression can be used to describe an ideal superconducting thin film as well, with m replaced by two times the electron mass, and with the superfluid fraction given by

$$n_s^{2D}(T) = n_s^{2D}(0)\lambda^2(0)/\lambda^2(T), \tag{3}$$

where λ is the penetration depth. With $n_s^{2D}(0) = d n_s^{3D}(0)$, n_s^{3D}being the 3D superfluid fraction, this captures the linear d dependence of T_φ, but taking n_s^{3D} as the 3D electron density in Pb predicts too large a value for T_φ. That is not surprising, however, given that the superfluid fraction at zero temperature is expected to be substantially suppressed by disorder [28], and indeed, it vanishes entirely as the 2D density is reduced further by decreasing d. The proximity to the superconductor–insulator quantum critical point suggests that it might be more appropriate to employ a scaling analysis [29,30] that shows that the superfluid fraction at zero temperature behaves as $n_s^{2D} \sim \delta^\zeta$ with $\zeta = (D + z - 2)\nu$ and $T_\varphi \sim \delta^{\nu z}$, where δ is the deviation from the quantum critical point, D is the dimension ($D=2$ in our case), z is the dynamical exponent, and ν is the correlation length exponent. Eliminating δ gives $T_\varphi \sim n_s^{2D}$ for $D=2$. Again, making the most natural assumption that the superfluid density scales with d explains the linear rise of T_φ with d. A direct measurement of the superfluid fraction will shed further light on the nature of the quantum phase transition [31].

We also note that no sign of BKT transition was seen in a recent *in situ* study of 1 ML In film superconductivity by Uchihashi et al. [32]. We believe that the reason is that their 2D system has a very low sheet resistance, and thus the BKT transition temperature T_φ is so high that it is pre-empted by gap closing. That underscores the crucial role played by the proximity to the superconductor–insulator quantum critical point in enabling the BKT transition in superconducting films. Superconducting transition was also reported in thin films of high temperature superconductors by Hetel et al. [31]. There is an interesting distinction from our study, however. In our systems, the reduced dimensionality is fundamental in causing phase fluctuations to destroy superconductivity; in high temperature superconductors, in contrast, phase fluctuations destroy even the 3D superconductivity, producing the so-called pseudo-gap phase with a nonvanishing amplitude of the order parameter. Sacépé et al. [33,34]. have also observed two distinct phase transitions in titanium nitride films, which they attribute to superconducting fluctuations.

4. Conclusions

Scanning tunneling microscopy and transport measurements on a series of crystalline ultrathin lead films demonstrate that Cooper pairing and superconductivity disappear at two distinct temperatures for films thinner than 9 monolayers, implying that an intermediate state contains Cooper pairs but they do not superconduct. The nonlinear transport properties of these thin films are consistent with the BKT model. The transition temperature of films between 4 and 9 monolayers is found to scale linearly with film thickness, suggesting a behavior governed by the proximity to superconductor–insulator quantum critical point.

Acknowledgments

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.ssc.2013.04.025.

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