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Little-Parks Oscillations in an Insulator

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We present the results of a magnetoresistance study of the disorder-induced superconductor-insulator transition in an amorphous indium-oxide thin film patterned by a nanoscale periodic array of holes. We observed Little-Parks-like oscillations over our entire range of disorder spanning the transition. The period of oscillations was unchanged and corresponded to the superconducting flux quantum in the superconducting as well as in the insulating phases. Our results provide direct evidence for electron pairing in the insulator bordering with superconductivity.

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A key ingredient in the Bardeen, Cooper, and Schrieffer theory of superconductivity is the pairing of electrons in the superconducting state [1]. One of the earliest [2] experimental supports for this prediction was obtained by Little and Parks (LP) [3], who measured the resistance (R) of a thin superconducting cylinder threaded by an external magnetic field (B). They observed magnetoresistance oscillations periodic in the superconducting flux quanta, \( \Phi_0 = \hbar / 2e \), where \( \hbar \) is Planck’s constant and \( e \) is the charge of the electron, corresponding to a Cooper-pair charge of 2e. LP-like oscillations were later observed in other, multiply connected, systems such as arrays of superconducting wires [4], arrays of Josephson junctions [5], and patterned thin superconducting films [6].

The transport properties of a superconducting material are strongly dependent on the interplay between superconductivity, electron-electron interaction, and disorder. In highly disordered, thin-film, superconductors this interplay can lead to a direct transition between the superconducting and insulating phases, for the review see Ref. [7]. This superconductor-insulator transition (SIT) can be induced by a variety of means such as disorder, film thickness, external magnetic field, external electric field, or chemical substitution [8–10].

Most (but not all, see Refs. [11,12]) of the theoretical attempts to account for the physics of the SIT [13–18] suggest that, surprisingly, Cooper-pairing plays a central role in determining the transport properties of the insulator bordering with superconductivity. Some of the authors [19] even claim that the insulating phase owes its very existence to Cooper-pairing. Although this notion is supported by circumstantial evidence, it still defies a direct verification.

In order to test for the role of electron pairing in the insulating phase, Stewart and his collaborators [20–23] conducted transport measurements of periodically patterned, amorphous, Bi thin films. The effect of the patterning of their Bi films was twofold. First, they observed LP-like oscillations in the insulating (and the superconducting) regime, with a period that was consistent with \( \Phi_0 \) threading a unit cell of their pattern. But as importantly, the patterning of their films resulted in a profound change to the phenomenology of their insulating phase, in the form of the appearance of the high-B insulating peak [7,24] that was absent in all their unpatterned samples [22].

In the Stewart et al. experiment, the SIT was driven by varying the film thickness. This resulted in variations in the geometry of the physical samples, as an unavoidable consequence of the topography of the substrate [23]. As a result of this, they claim, the insulator bordering superconductivity consisted of a network of superconducting islands connected by weak links of nonsuperconducting material [25]. They suggested that this artificially introduced inhomogeneity resulted in a transition that was dominated by phase, rather than amplitude [21], fluctuations in the order parameter, in turn leading to the formation of a bosonic, or Cooper-pair insulating state, rather than the fermionic insulator they observed in the unpatterned films. This change in the microscopic physics of the transition, they argued, was responsible for the change in the phenomenology in the insulating regime and the appearance of the insulating peak.

In this Letter we present the results of transport measurements of a nanopatterned, amorphous indium-oxide (\( a:\text{InO} \)) thin film. By means of a simple annealing procedure (see below) we were able to affect a SIT driven by the amount of disorder in the film [26]. We observed magnetoresistance oscillations with a period corresponding to \( \Phi_0 \), which remained unchanged as we went from the insulator to the superconducting phases. In contrast with the work on Bi thin films where the magnetoresistance peak was only observed in patterned films, our procedure enabled the SIT in a single physical sample without any change in geometry and without inducing a change in the phenomenology associated with the bosonic insulating state that exists in the absence of any patterning [see
The hexagonal lattice of differential preamplifier and measured using an EG&G
the sample was amplified by a homemade, low-noise, measured using a four-probe configuration with currents in
rents in the range
transport measurements. For the insulating samples, a
(Oxford Instruments Inc.) dilution refrigerator for the
holder and electrically connected by Au wire using silver
after evaporation the sample was mounted on the sample
shadow mask 3 mm long and 1.5 mm wide. Immediately
appearing before the large magnetoresistance peak.
Analyzing scanning electron microscope images we found
a hole diameter of
Analyzing scanning electron microscope images we found
a hole depth, the hole pattern transferred to the
substrate. The growth condition of the oxide layer
resulting in a unit-cell area of
Ω/  nA
FIG. 1 (color online). (a) R vs T at B = 0 T in annealing
states A through K, spanning the range of disorder in nano-patterned a: InO thin-film sample. (b) R vs B in superconducting
state J at T ≈ 0.03, 0.04, 0.06, 0.08, 0.1, 0.15, 0.2, 0.3, 0.35, 0.45,
0.55, 0.6, 0.7, 0.8, 0.9 K . Inset: Critical B vs R at room T
for some of the superconducting states. The dashed line is a
linear fit.
After completion of the low-T measurements, the sample
was annealed at 43 ± 3 °C while maintaining a vacuum of
~3 × 10⁻² Torr, and then measured again at low-T.
In Fig. 1(a) we show R vs T taken at B = 0 T for several
annealing states spanning our range of disorder. Our a: InO
film was initially in an insulating state, attested to by its
diverging R as T → 0. The annealing (we label each annealing
state in alphabetical order) resulted in weaker insulators and, subsequently, superconductivity com-
enced with state H. We found that the R(T) traces in the
insulating phase followed an Arrhenius law R(T) ∝
exp(T₀/T) where T₀ is the activation T [24]. With each
annealing state, T₀ was found to decrease. Unfortunately,
in this experiment, we have taken a large annealing step on
the approach to superconductivity so we were not able to
determine the critical normal-state R (at T = 0.9 K) of the
disorder-driven SIT to better than specifying that it is in the
range of 2 < R(kΩ/□) < 8. In the superconducting re-
gime, the critical T increased from 0.4 K in state H to
0.9 K in state K. A much more detailed account of this
annealing-driven SIT is forthcoming.
In superconducting samples, the resistance was strongly dependent on the
magnetic field, especially around B = 0 T. Meaning that
applied magnetic fields of a few mT caused resistance to
increase by many orders of magnitude. Therefore, the R(T)
dependence for the superconducting samples was recon-
structed from R(B) data measured at fixed T’s. The value of
R(B = 0) was identified as the minimum value observed in
a range −0.1 < B(T) < 0.1.
We begin the presentation of our B-dependent data by
plotting, in Fig. 1(b), the R isotherms obtained from one of
our superconducting states, state J, over our entire B range.
Before addressing the signature of the patterning in our
data evident at B < 1.5 T, we note that our patterned
sample exhibits the familiar [24] high-B phenomenology
that we are accustomed to in our previously studied a: InO
films. Typical electron concentration in such films is on the
order of 10²⁰ cm⁻³ with an upper critical field of about
Hc₂ = 14 T. This includes the crossing point of the iso-
therms at B = 1.5 T identified with the critical B, Bc, of
the B-tuned SIT, followed by the prominent magnetoresis-
tance peak at B = 7 T. In the inset of Fig. 1(b), we plot Bc
in the superconducting phase at various annealing states
versus R at room temperature, RRT, which we take as a
rough measure of the level of disorder in our sample. A
clear trend was detected in which Bc is decreasing with
RRT and the extrapolated vanishing of Bc is expected at
RRT = 3.7 kΩ. The clear evidence for the applied nano-
pattern, was the magnetoresistance oscillations, observed
in the superconducting regime below B = 1.5 T.
In Fig. 2, we plot R vs B for our most superconducting
state K, measured at T = 0.08 K, in the range of
−1 < B(T) < 1. Nearly four oscillations are seen,
superimposed on a rising background. The oscillations
period, 0.35 ± 0.005 T, corresponds to Φ₀ threading an
area of 5500 nm², that is, within error, the unit-cell area of our AAO substrate. In the inset of Fig. 2 we present \( R(B) \) isotherms, obtained from the same state, in the range of 0.03 < \( T(K) < 0.9 \) on a logarithmic scale. The oscillations period is independent of \( T \), indicating that it is determined by the geometry of the pattern rather than by some other physical length scale, which is expected to be \( T \) dependent. Following Refs. [4,6] we interpret these results as the LP oscillations.

Our central finding presented in Fig. 3(a), where we plot \( R(B) \) traces of our sample spanning our entire range of disorder. Clear oscillations are observed throughout the range and, most importantly, the period of oscillations remains fixed, corresponding to \( \Phi_0 \) through a unit cell of the array, deep into the insulating phase. While in this figure we show data at \( T = 0.55 \) K because, at lower \( T_s \), the insulating states have prohibitory high \( R_s \), lower-\( T (= 0.15 \) K) data are plotted, for a superconducting state \( I \) and an insulating state \( F \) in Fig. 3(a) [the corresponding \( B = 0 \) \( R \) vs \( T \) can be seen in Fig. 1(a)]. Strikingly, the amplitude of the oscillations in the insulating state can be very large, see Fig. 3(b). At \( T = 0.15 \) K, for state \( F \) it was \( 10^8 \) \( \Omega/\square \) and grows even larger at lower \( T_s \). The observation of these oscillations demonstrate phase coherence between Cooper pairs in the insulating regime on a scale larger than the interhole distance. So far, we discussed the \( B \) dependence of the resistance near zero \( V \) bias. However, a similar oscillatory trend was observed under application of a finite \( V \), where the electronic transport in the insulating states is characterized by high nonlinearity [29]. This is demonstrated in Fig. 4, in which \( I - V \) characteristics of insulating state \( A \) at \( B = 0 \) T are presented. Similarly to the nonpatterned films case, at low \( T \) these \( I-V \) curves exhibit an abrupt jump in \( I \) of several orders of magnitude at a threshold dependent on the sweep direction. On increasing \( V \), the system switches from a high-resistance to a

![Fig. 2](color online). \( R \) vs \( B \) at \( T = 0.08 \) K for superconducting state \( K \). The period of LP oscillations was \( 0.35 \pm 0.005 \) T. Inset: \( R \) isotherms taken at various \( T \)’s in state \( K \).

![Fig. 3](color online). (a) \( R \) vs \( B \) from different annealing states at \( T = 0.55 \) K spanning our entire range of disorder. The states \( A \) through \( G \) were insulating, while states \( H \) through \( K \) were superconducting. (b) \( R \), plotted on a logarithmic scale, vs \( B \) for insulating state \( F \) and superconducting state \( I \). The dashed line corresponds to \( \Phi_0 \) penetrating an area of one unit cell.

![Fig. 4](Nonlinear \( I - V \) in insulating state \( A \) at \( T = 0.025 \) K at three different \( B \)’s. Arrows indicate direction of voltage sweep. Inset: threshold voltages \( V_{LH} \) and \( V_{HL} \) vs \( B \)).
low-resistance state at a point referred to as $V_{HL}$. A hysteresis is observed when the sweep direction is reversed, until a second switching voltage, $V_{HLH}$, is reached. Tracing these thresholds as a function of $B$, we observe (inset in Fig. 4) oscillations with period $\Phi_0$ superimposed on a monotonically increasing background. These oscillations appear to reflect the periodic oscillations observed in the Ohmic measurements. In some of the superconducting states multiple crossing points [21] of the isotherms were observed, see Fig. 5. $R_s$ at the crossing points were found to be close to $R = 4.2 \, \text{k}\Omega/\square$, similar to other superconducting states [9,24]. For state $H$ at $B > 0$ T there were three such points at $B^* = 0.06$ T, $B^* = 0.31$ T, and $B^* = 0.39$ T. As $B$ increased from zero, the superconducting sample became insulating at $B^* = 0.06$ T. At half filling $B = 0.18$ T the $R$ was larger then $10^7 \, \Omega/\square$. The sample returned to the superconducting state at $B^* = 0.39$ T. However, its minimum $R$ at $B = 0.35$ T was larger than at $B = 0$ T. Above $B = 0.39$ T, the insulating state reappeared. With the increased annealing time, the sample became more superconducting and the critical $B^*$ shifted to the higher values.

In summary, we observed LP-like oscillations in the insulating and superconducting phases of a single physical sample of nanopatterned $\alpha$-InO thin-film. The $T$-independent oscillations had a constant period throughout the disorder-tuned SIT. For our sample, the patterning induced no significant change in the high-$B$ magnetoresistance. The flux periodicity corresponded to $\Phi_0$ through the unit cell of the patterned array, demonstrating the participation of Cooper pairs in the transport in the insulating phase.

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