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On the origin of the anomalous sign reversal in the Hall effect in Nb thin films

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We re-visit the anomalous sign reversal problem in the Hall effect of the sputtered Nb thin films. We find that the anomalous sign reversal in the Hall effect is extremely sensitive to a small tilting of the magnetic field and to the magnitude of the applied current. Large anomalous variations are also observed in the symmetric part of the transverse resistance R_{xy}. We suggest that the surface current loops on superconducting grains at the edges of the superconducting thin films may be responsible for the Hall sign reversal and the accompanying anomalous effects in the symmetric part of R_{xy}.

Keywords: anomalous sign reversal, Hall effect, edge supercurrents, Aharonov–Bohm effect

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1. Introduction

In thin film superconductors, the transport properties associated with the resistive transition in a magnetic field have been of longstanding interests. An unresolved controversy is the origin of the anomalous sign reversal in the Hall effect, first debated in 1990s,[1–8] and it has generated great controversies recently.[9–11] Several possible mechanisms have been proposed.[2–6,10] However, in a study of disordered superconducting tantalum nitride films,[12] no such sign reversal was observed in the Hall effect across the resistive transition in a magnetic field. Thus it remains a mystery as to the origin of the observed anomalies in the Hall effect in the systems such effects have been observed.

In this study, we wish to re-visit this problem in sputtered niobium (Nb) thin films, a system that was investigated previously and an anomalous sign reversal in the Hall effect was observed under certain conditions.[7] Recently, it has been suggested[13,14] that much of the broadening in the resistive transition from the normal state to the superconducting state in sputtered Nb thin films is due to the surface superconductivity. Thus whether the previously observed anomalous sign reversal in the Hall effect is related to the surface superconductivity in this system is an unresolved question.

Edge or surface superconducting state was predicted long ago by Saint-James and de Gennes (SJdG)[15] and this effect has been confirmed in bulk samples with a flat surface that one can align the magnetic field with.[16,17] The surface superconductivity was also believed to play a role in the vortex physics in the bulk between upper critical field $H_{c2}(T)$ and surface nucleation field $H_{c3}(T)$.[18,19] For a thin film superconductor with a magnetic field applied normal to the film, the surface superconductivity effects have largely been ignored in the past. However, two recent experimental studies[13,14] clearly suggested that the broadened resistive transition can be, at least partially, attributed to the surface superconductivity of the crystalline Nb grains with their surfaces being parallel to the magnetic field.

Here we report an experimental study of the Hall effect and the magneto-resistive transition in sputtered Nb thin films. We find that, as previously reported,[7] there is indeed an observable sign reversal of the Hall effect across the normal-superconducting transition as a function of the applied magnetic field. We find that this anomalous sign reversal in the Hall effect appears or disappears depending on a small change of the field orientation relative to the edge surface of the sample. A simple model is proposed that the anomalous sign reversal of the Hall effect may be caused by the edge surface supercurrents on the superconducting grains at the edges, due to the surface superconductivity.

2. Materials and methods

The materials used in this experiment are Nb thin films, of nominal thickness of 120 nm. The details of the Nb thin film deposition and characterization were described elsewhere.[20,21] As shown in Fig. 1(a), the sample region consists of a 1 mm long horizontal channel with a width of 10 μm, the vertical lines are used for longitudinal and transverse voltage probes, with a width of 5 μm. The sample was patterned

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using standard photolithography and etched into the final form using the ion beam etching (IBE) process.[22] The surface of the Nb film Hall device was checked by the scanning electron microscopy (SEM), Figs. 1(a)–1(d). On the length scale above 100 nm, the film looks smooth and the IBE etched sample edges are sharp. However, below 100 nm, one can observe the poly-crystalline nature of the film, as shown in Fig. 1(d). We believe that the nanoscale roughness of the edges is the key to the understanding of the quantum transport of edge supercurrents. The transport measurements were carried out using the standard four-probe method with a commercial measurement system (Quantum Design PPMS-9). Alternating current directions were used to eliminate the thermal electric signals.

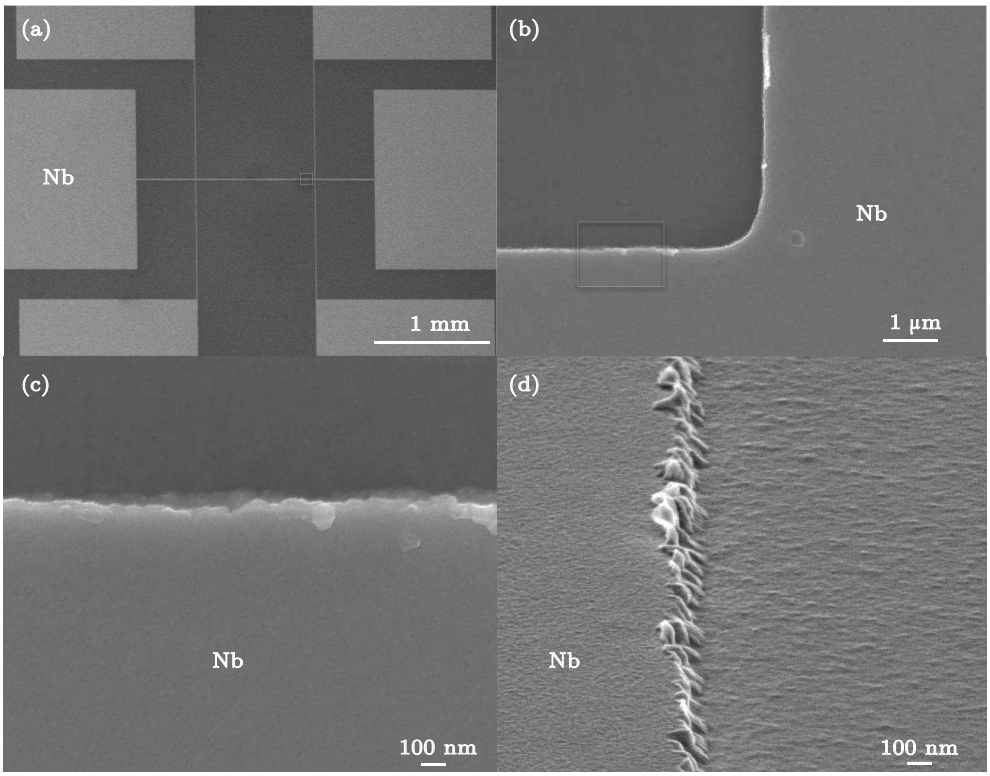


Fig. 1. SEM micrographs of a patterned Nb thin film sample. (a) Whole sample, scale bar: 1.0 mm. (b) The expanded view of the small box region in (a); scale bar: 1.0 μm. (c) The expanded view of the small box region in (b); scale bar: 100 nm. (d) A close-up view of a part of an edge; scale bar: 100 nm.

The as-sputtered Nb thin-film sample used in this experiment has a superconducting transition temperature at $T_c = 8.875$ K in zero magnetic field. The superconducting transition temperature is defined as the temperature at which the sample resistance has decreased to 50% of the normal-state value at 10.0 K. Using the normal-state resistivity ρ_{xx} at 10 K, we estimate the Drude mean free path $l = \hbar k_F/ne^2\rho_{xx}$ to be 18.5 nm, or about the grain size of the sputtered Nb films, see Fig. 1.

3. Results and discussion

Figure 2 shows the longitudinal and transverse resistances for the superconducting–normal transition at 4.0 K, as a function of magnetic field H, at $I = 100$ μA, $T = 4.0$ K, and at three different angles between the sample and the magnetic field, as illustrated in the inset in Fig. 2(a). The "no-rotation" configuration is when the magnetic field is perpendicular to the film (thus parallel to the edge surfaces, within 1°–2°). The longitudinal resistance R_{xx} and transverse resistance R_{xy} are defined as $R_{xx} = V_{xx}/I_x$ and $R_{xy} = V_{xy}/I_x$, respectively. Since the voltage signals due to the Hall effect change sign when the magnetic field direction is reversed, we plot both the symmetric and anti-symmetric parts of R_{xx} and R_{xy}, as shown in Figs. 2(a)–2(d).

Figure 2(a) shows the symmetric part of R_{xx}, $(R_{xx}(H) + R_{xx}(-H))/2$, which is the longitudinal resistance along the 1 mm long path. The transition from zero resistance superconducting state to the normal state is broad, over 3 kOe. We defined H_{c2} as the steepest slope in $R_{xx}(H)$ and H_{c3} as where the longitudinal resistance first starts to decrease ($\sim 1\%$ change) from the normal state value. This broadened resistive transition in Nb attracted attention recently[13,14] with respect to the surface superconductivity. Tilting the sample with respect to the magnetic field has negligible effects in this longitudinal resistance.

However, as shown in Fig. 2(b), tilting the magnetic field has a drastic effect on the anti-symmetric part of R_{xx}, $(R_{xx}(H) - R_{xx}(-H))/2$. When the sample is tilted either around x or y axis, R_{xx} (anti-symmetric) changes from two

small peaks around H_{c2}, for field normal to the sample, to a large peak as a function of the magnetic field when the sample is tilted by 10°, only tending to zero at H_{c3}. The asymmetry effect of the sample geometry imperfection is small in our device and is not the cause of the effect here.

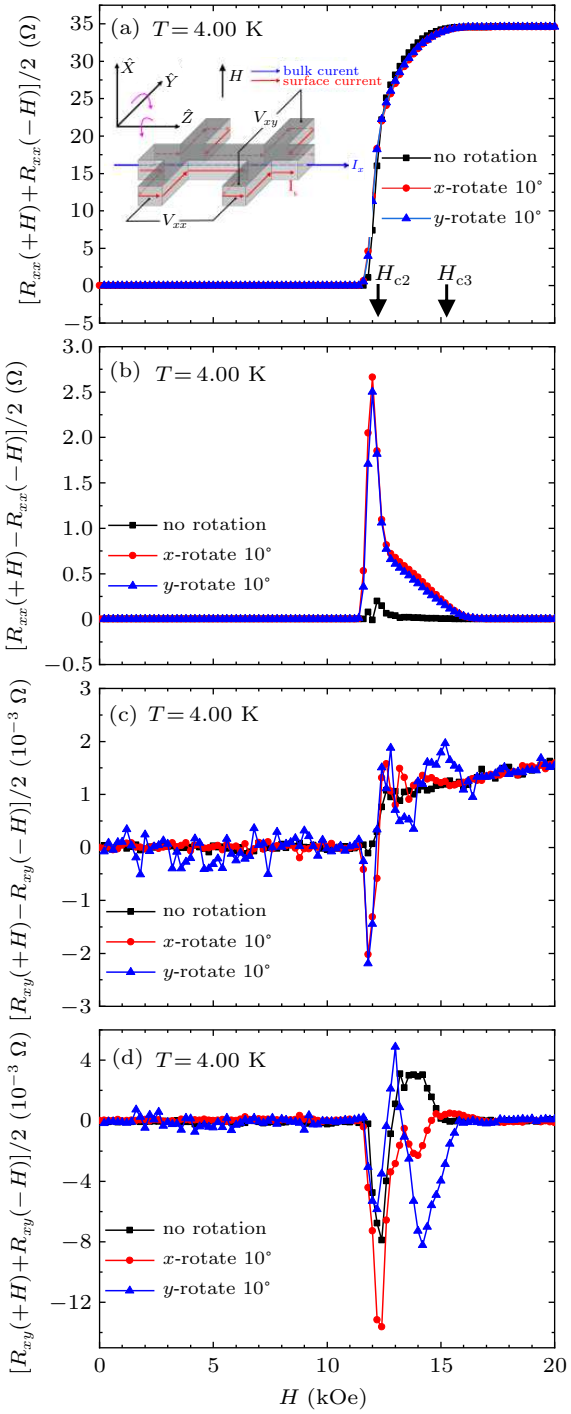


Fig. 2. Magnetic field dependence of longitudinal and transverse resistances. (a) The symmetric part of the longitudinal resistance R_{xx}. (b) The anti-symmetric part of the longitudinal resistance. (c) The Hall resistance, the anti-symmetric part of the transverse resistance. (d) The symmetric part of the transverse resistance. $I = 100$ μA, $T = 4.0$ K.

The anti-symmetric part of the transverse resistance, R_{xy}, $(R_{xy}(H) - R_{xy}(-H))/2$, is the Hall effect, as shown in Fig. 2(c). When the magnetic field is perpendicular to the film surface, with decreasing field, the Hall resistance is a linear function of the magnetic field down to H_{c2} where it drops to zero, with no sign change. However, when the sample is rotated by 10°, either around x-axis or y-axis, (see Fig. 2(a) inset for clarity), there are oscillatory behaviors in the Hall resistance. The Hall resistance clearly changes sign at H_{c2}. Similar oscillatory behavior in R_{xy} was reported in Ref. [7], but was erroneously attributed to the vortex pinning behavior in the vortex state since the H_{c3} was misidentified as the H_{c2}.

The symmetric part of the transverse resistance R_{xy} is resistive since it is due to a longitudinal voltage developed along the direction of the current. Figure 2(d) shows large oscillations as a function of the magnetic field for all three field configurations. To be discussed in detail below, we believe these oscillations are due to the Aharonov–Bohm interference effects around the nanoscale grains on the edges of the voltage probes.

A simple way to test if the anomalies in Fig. 2 are due to the surface superconductivity is to check how they depend on the amplitudes of the applied currents. The surface superconducting layer cannot support a large current since it flows within a thin region of the size of the coherence length and it is limited by the depairing current density. Figure 3 shows the effects of the applied current in R_{xy} vs. H. We find that the anomalous effects in Fig. 2 are extremely sensitive to the magnitude of the applied current. For clarity, the anti-symmetric (Hall effect) and the symmetric parts (resistive) of R_{xy} data are plotted in an expanded view, zooming into the transition region. With decreasing field, when H_{c3} is crossed, the anti-symmetric part of R_{xy} shows the normal Hall effect and drops to zero at H_{c2}, while the symmetric part of R_{xy} exhibits a large increase at low currents 100–200 μA, this effect is suppressed at 500 μA, demonstrating that these features are indeed surface current effects.

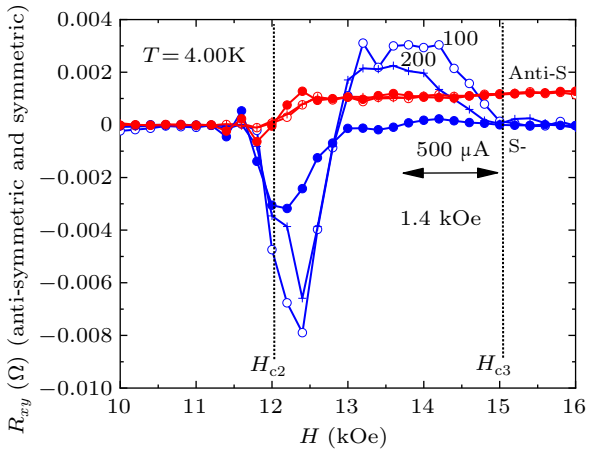


Fig. 3. Effects of applied current: anti-symmetric (red, Hall effect) and symmetric parts (blue, resistive) of the transverse resistance R_{xy}, at $T = 4.00$ K, $I = 100$, 200, 500 μA. R_{xy}(Anti-S) $= (R_{xy}(H) - R_{xy}(-H))/2$, R_{xy}(S) $= (R_{xy}(H) + R_{xy}(-H))/2$. Open circles, crosses, and solid circles are for 100 μA, 200 μA, and 500 μA respectively. The 100 μA data are the same as the "no rotation" in Fig. 2 for reference.

The next question is how these anomalous behaviors change with increasing temperature. Figure 4 shows the magnetic field dependence of the transverse resistance at 4–9 K. Two major features can be discerned immediately. First we notice that the oscillatory behavior between H_{c2} and H_{c3} fades away with increasing temperature. Second, which is a surprise, the symmetric part of R_{xy} changes abruptly from a large negative peak near H_{c2} to a large positive peak, when temperature is increased from 5 K to 6 K, then back to a negative peak again from 7 K to 8 K. This behavior cannot be explained by the pinning behavior in the vortex state, as often attributed to in the literature.[2–4,7,9,11] It can be easily accounted for in the model we present below.

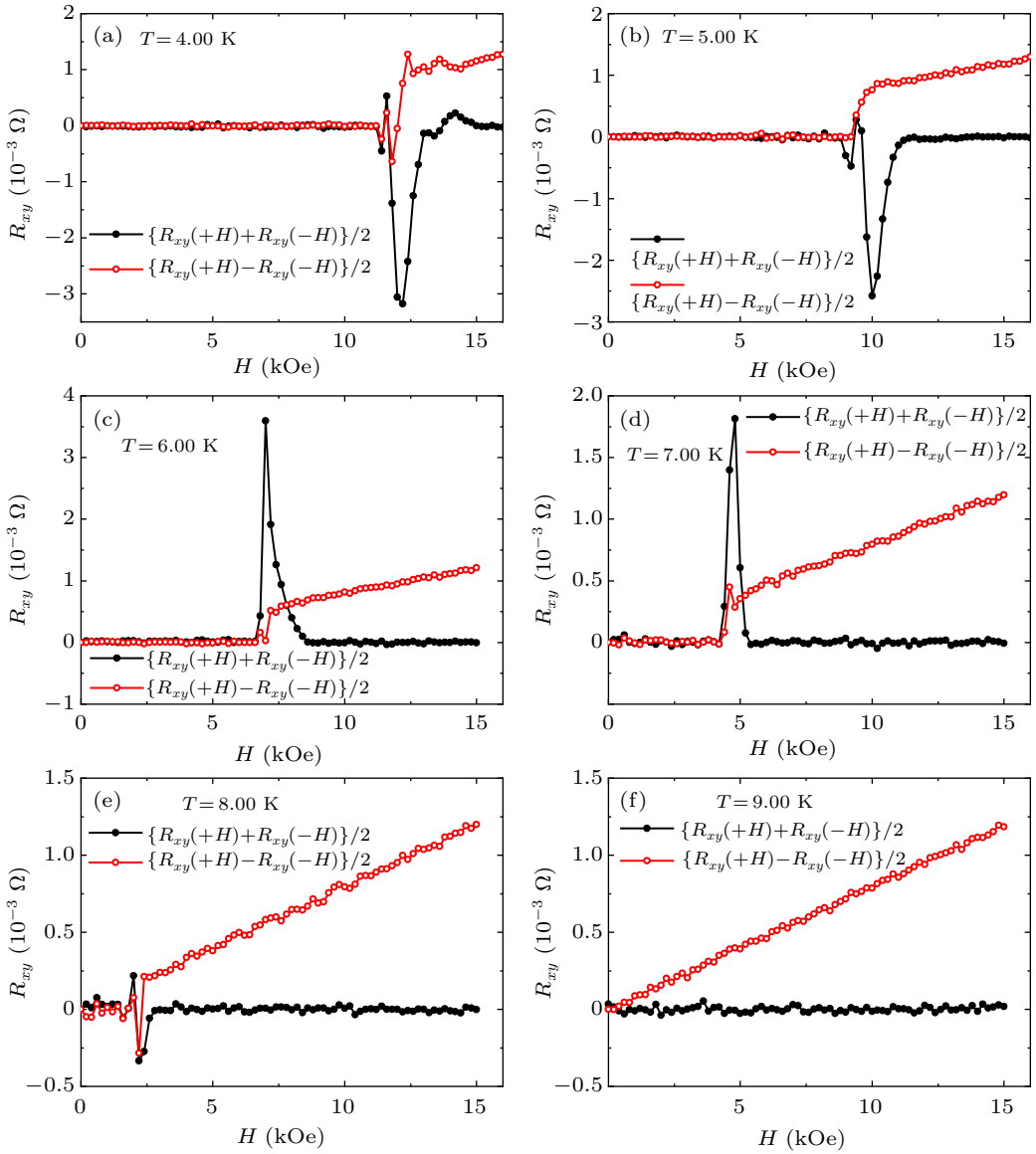


Fig. 4. Magnetic field dependence of transverse resistance R_{xy} at temperature (a) 4 K, (b) 5 K, (c) 6 K, (d) 7 K, (e) 8 K, (f) 9 K, respectively. For this sample, the zero-field T_c(half normal-state resistance) = 8.875 K. The anti-symmetric part is the Hall resistance, the symmetric part is the anomaly discussed in the text.

4. Model: edge supercurrent loops

In Fig. 5, we propose a simple model that provides a qualitative explanation for all of the observations here. Our model is based on the following observations. First, the resistance oscillations in Figs. 2 and 3 have a period of $\Delta H \sim 1.4$ kOe (as marked in Fig. 3). This field interval corresponds to a change of one flux quantum ($\Delta\Phi = 2.07 \times 10^{-7}$ G·cm^2, in CGS units) in the cross-sectional area of 1.48×10^{-10} cm^2, or (120 nm)^2. This suggests that the source of current loops is on the scale of 120 nm, not due to the current loop around the entire sample. We should emphasize that the signal is not a simple sinusoidal function of the magnetic field, the data show large variations with the magnetic field. We envision that each grain has its own oscillatory dependence and what we observed is an additive effect of many Aharonov–Bohm oscillations.

Second, a close examination of the SEM micrographs (Fig. 1(d)) will convince us that on the scale of $\sim$ 100 nm, the Nb film edges are quite rough. Since an Aharonov–Bohm ring

attached to a 1D conduction channel can change the transmission rate in the channel,[23] given the poly-crystalline nature of the sputtered Nb films, it is reasonable to assume that there are many 100 nm scale grains along the edges that are capable of acting as microloops of supercurrents with Aharonov–Bohm interference effects, which will result in a modulation of resistance in a constant current measurement. In Fig. 5, we draw two such nanoscale particles, or grains, for illustration. Since clearly there is a distribution of grain sizes, one expects a distribution of Aharonov–Bohm periodicities. After a closer examination of the curves in Fig. 3 one can see that the modulation effects of multiple periods in magnetic field are indeed visible.

We should point out that the Drude mean free path of the normal-state electrons in this sample is of the order of 18.5 nm, thus the observed field-dependent resistance oscillations cannot be due to the phase-sensitive behavior of the normal electrons. The fact that the effects occur between H_{c3} and H_{c2} strongly suggests that it is caused by the surface supercurrents. Recently Aleiner *et al.*[24] discussed the possibility of this behavior.

The first anomaly that can be accounted for by the model in Fig. 5 is the anomalous peak in the anti-symmetric part of the longitudinal resistance R_{xx}, as shown in Fig. 2(b). This is a pseudo Hall effect. It is larger than the real Hall effect, in Fig. 2(c), by 3 orders of magnitude. Thus it cannot be explained by the imperfection in the Hall probe geometry. It can be naturally explained if we realize that there is a diamagnetic surface current (the blue dashed lines in Fig. 5) due to the edge surface superconductivity, as it is implied in the SJdG theory.[15] This SJdG diamagnetic current reverses direction when the external magnetic field is reversed. Thus the edge currents on the L_1 and L_2 paths are not equal. This inequality in edge current changes sign when the field is reversed, resulting in a pseudo Hall effect, the antisymmetric signal in the longitudinal resistance R_{xx}.

It should be noted that the diamagnetic current in the SJdG surface superconductivity theory is an equilibrium diamagnetism, not the induced screening current due to a field change. This can be tested by comparing the results for field-increasing and field-decreasing runs. Our results (not shown) show that they are identical, i.e., reversible, thus ruling out this scenario.

The model in Fig. 5 also offers a self-consistent explanation for the oscillations observed in the transverse resistance R_{xy}. The anti-symmetric part of R_{xy}, Fig. 2(c), is the Hall effect; the symmetric part of R_{xy}, Fig. 2(c), is a longitudinal, resistive signal, a voltage along the current path. For magnetic field normal to the film, the "no rotation" mode, with decreasing H, the Hall resistance follows the normal state value ($\sim H$) down to H_{c2} where it drops to zero with no sign change. However, the symmetric part of R_{xy} exhibits large oscillations, starting at H_{c3} and ended at H_{c2}, with decreasing magnetic field. When the sample is rotated by 10°, around either x-axis or y-axis, large oscillations appear in both anitisymmetric (Hall) and symmetric (resistive) parts of R_{xy}. These anomalies can easily be explained if we assume there is a nanoscale grain, which can support an Aharonov–Bohm current loop, on the edge of C_1 edges of the C voltage probe. Combined with the diamagnetic current that changes sign when the magnetic field is reversed, it is easy to see that an Aharonov–Bohm effect modulated resistance will give rise to both a pseudo Hall effect and a longitudinal resistance signal in the R_{xy}. In our model, the anomalous sign reversal of the Hall effect in Fig. 2(c) is a pseudo Hall effect due to the edge supercurrent, it has the same origin as the anomalous peak in the anti-symmetric part of R_{xx} in Fig. 2(b), i.e., the diamagnetic current due to surface superconductivity which breaks the symmetry in the edge currents on the opposing edges of the sample.

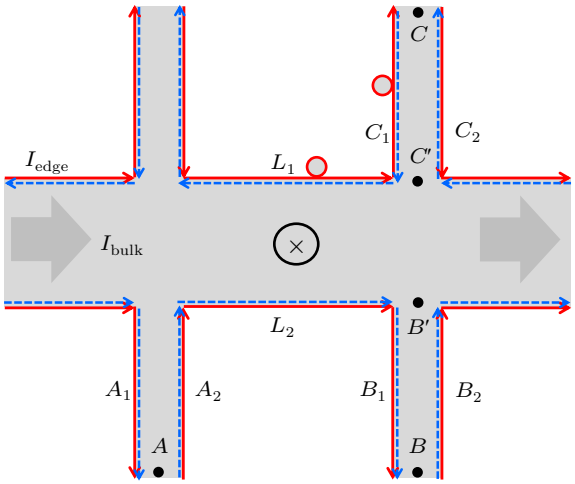


Fig. 5. A qualitative model of edge supercurrents in a Hall bar geometry with nanoscale grains, attached on the C_1 side of transverse voltage probe C and on the L_1 side of the main channel (not to scale). The applied current is from the left terminal to the right. When the applied field is between H_{c2} and H_{c3}, the bulk normal current flows from left to right through the horizontal channel. The red lines are the edge supercurrent from the current source, the blue dashed lines are the diamagnetic current induced by the magnetic field in the surface superconducting state. The diamagnetic currents reverse directions when the external magnetic field is reversed. In contrast, the edge currents due to the applied current do not change directions when the magnetic field is reversed.

Finally, our model can account for the temperature dependence data in Fig. 4 as well. As shown in Fig. 4, with increasing temperature, most of the resistance oscillations between H_{c2} and H_{c3} disappear, as expected for the Aharonov–Bohm effect since the surface current loses its phase coherence. The change from a large negative peak to a positive peak, in Fig. 4, can be understood if we consider the temperature dependence of the superconducting coherence length. With the coherence length increasing, the effective size of the superconducting particles will be changing. Effectively, the local current loop in C_1 may disappear and new ones may emerge on the edge

of B_1. When that happens, the symmetric part of the voltage between probes B and C will change sign.

5. Conclusions

In summary, we have re-visited the anomalous sign reversal problem in the Hall effect in the system of sputtered Nb thin films. We find that the anomalous sign reversal in the Hall effect is extremely sensitive to a small tilting of the magnetic field and to the magnitude of the applied current. Large anomalous variations are also observed in the symmetric part of the transverse resistance R_{xy}. We suggest that the observed anomalies may be accounted for if there are surface current loops on superconducting grains at the edges of the superconducting thin films. We should point out that our data are consistent with the simple model proposed in Fig. 5, but they are not definitive proof. An ideal experimental proof of the proposed model in Fig. 5 would be using amorphous films and lithographic techniques to create a structure as shown in Fig. 5. With such a structure, one can directly correlate the Aharonov–Bohm effect with the diameter of the islands.

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