PHYSICAL REVIEW B **81**, 100503(R) (2010)

Fluxoid quantization in the critical current of a niobium superconducting loop far below the critical temperature

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(Received 30 June 2009; revised manuscript received 1 February 2010; published 5 March 2010)

The critical current of a niobium type II superconducting loop has been measured far below the critical temperature using contacts applied symmetrically or asymmetrically around the loop. The magnetic field dependence of the critical current allows a direct observation of the circulating current resulting from the fluxoid quantization and presents a saw-tooth behavior. The periodicity is given by a flux quantum through the outer area of the loop. Despite the presence of many vortices at high magnetic fields, the circulating current finds its way in between them and still has an impact on the total critical current of the loop. Macroscopic quantum coherence effects in such a niobium loop allow observing single quanta changes of the fluxoid up to a magnetic field of 1.2 T.

DOI: 10.1103/PhysRevB.81.100503 PACS number(s): 74.78.Na, 74.25.Sv

A classical superconducting quantum interference device (SQUID) consisting of a macroscopic superconducting loop interrupted by one or two Josephson junctions allows to reach the impressive resolution of 1 fT. Fink *et al.*[1] proposed another type of SQUID based on a homogeneous junction free, mesoscopic superconducting loop, in which the critical current varies with the applied magnetic field also in an oscillating manner. The interest for such loop resides also in their possible use as flux qubit,[2]where decoherence induced by the junction is suppressed. An experimental realization of this type of SQUID has been reported in the case of mesoscopic aluminum loop very close to the critical temperature T_c.[3] Indeed, it was often postulated that such a manifestation of quantum coherence was restricted to overall loop size that are smaller than the superconducting coherence length ξ (as it is the case close to T_c) and to very narrow arm width. Here we show that far from T_c, homogeneous loop made of niobium, which is a type II superconductor with a very small coherence length compared to the loop dimensions, still displays macroscopic quantum coherence effects. Whereas critical current oscillations are limited below about 10 mT for aluminum loop, here the vortex penetration allows to observe the critical current oscillations up to a magnetic field of 1.2 T.

The quantization of the fluxoid Φ' (rather than the flux Φ) in a multiply connected superconductor is a well established phenomenon.[4,5] Indeed, using Bohr's quantum condition the fluxoid can be expressed as

$$\Phi' \equiv \frac{\oint \vec{P} \cdot \vec{dl}}{q_{CP}} = \frac{nh}{2e} = n\Phi_0 \tag{1}$$

where $\vec{P}$ and $q_{CP}=2e$ are the momentum and the charge of Cooper pairs, h is the Planck constant, n an integer and $\Phi_0 \equiv \frac{h}{2e} = 2.07\times 10^{-15}$ T/m^2 is the flux quantum. The circulating current I_p impacts the critical temperature T_c due to the increase of kinetic energy (proportional to I_p^2) and it therefore leads to the well-known parabolic oscillations of the resistance $R(\Phi/\Phi_0)$ close to T_c.[5] Although this change in T_c is small, as small as 3 mK in the original Little-Parks experiment on a thin-walled tin cylinder, its observation is made easier by the large variation of the resistance due to the sharp superconducting transition. Since $\Delta T_c/T_c$ (where ΔT_c is the transition width) is proportional[6] to $(\xi/r)^2$, aluminum loops with large ξ and small radius r have often been used,[7,8] leading to $\Delta T_c \sim 20$ mK. Recently, small size superconducting loops have been the object of a renewed interest both theoretically[9] and experimentally with indirect[10–12] as well as direct (but close to T_c) (Refs. 13 and 14) measurements of $I_c(\Phi/\Phi_0)$ reported mainly for aluminum loops.

In this Rapid Communication we report on the direct observation of a saw-tooth behavior of $I_c(\Phi/\Phi_0)$ far from T_c for a niobium type II loop without any Josephson junction. Reproducibility has been tested on another similar sample. This oscillating behavior is observed at low magnetic field as well as at high magnetic field where vortex penetration is important. We have also explored the effect of an asymmetric biasing of the loop. Our observations of fluxoid quantization effects in strong type II superconducting loops far from T_c constitute an interesting development in multiple connected mesoscopic superconductors.

The typical layout of the loops investigated is shown in the inset Fig. 1. A 75 nm Nb film is deposited by dc magnetron sputtering on a Si/SiO_2 substrate. It is then patterned to form the loop by SF_6 reactive ion etching using a 20-nm-thick Al_2O_3 mask. The multicontacts are thus of the same nature than the loop under study and are labeled from 1 to 4. Each of these superconducting contacts splits up in two (not shown) so that a pseudo four contacts arrangement (two current plus two voltage lines) can be made for any given contact pair. Symmetric bias consists of using contacts 1 and 4, while asymmetric bias involves e.g., contacts 1 and 2. The

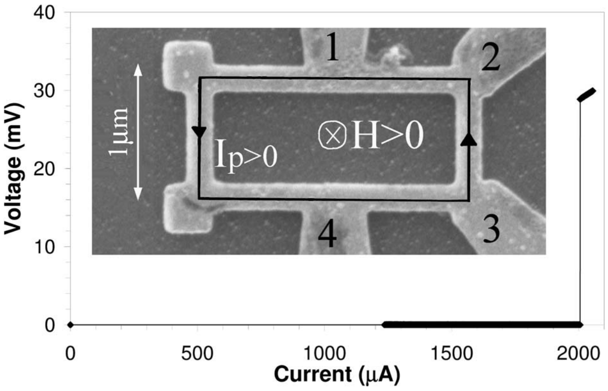

FIG. 1. Scanning electron micrograph of a 75 nm Nb loop. The position of the leads and the positive direction of the magnetic field H and the circulating current I_p are indicated. Also shown is a typical current-voltage characteristic at 1.5 K.

normal-state resistivity is 10 $\mu\Omega$ cm at 9 K, and the ratio between resistances $R(300\ \mathrm{K})/R(9\ \mathrm{K})=2.7$. From the free-electron model,[15] the electron mean-free path is estimated to be 9 nm, giving a low-temperature penetration depth $\lambda_0 \approx 90$ nm and a coherence length $\xi_0 \approx 15$ nm. A pulsed-tube cryocooler at a base temperature of 1.5 K was used with a combination of homemade RC and LC low-pass filters operative up to 40 GHz on every line. As shown on the current-voltage characteristic on Fig. 1, the critical current can be unambiguously determined (with an error bar of 1 μA) thanks to the abruptness of the superconducting transition.

Figure 2(b) shows the low magnetic field evolution of the critical current in the symmetrical case measured in two different configurations: for each value of the positive magnetic field (see Fig. 1 for the sign definition), the critical current is first measured by injecting a current $I_{4-1}>0$ (i.e., directed from 4 to 1, red points) and then it is measured again with the current in the opposite direction ($I_{4-1}<0$, blue squares).

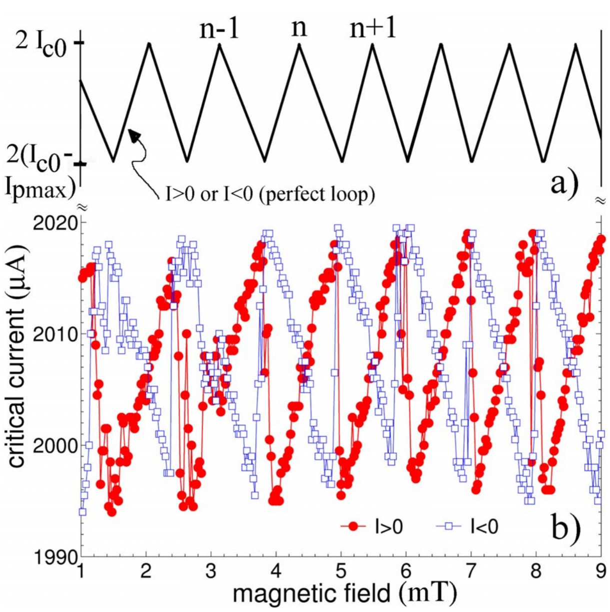

FIG. 2. (Color online) Critical current vs magnetic field at 1.5 K of a Nb loop measured with symmetric leads. (a) Sketches of the expected result for a perfect loop having the same switching current in each arms. (b) For each value of the (positive) magnetic field, the critical current is subsequently measured in the positive (red dot) and negative (blue square) current direction (current flowing from leads 4 to 1 and then 1 to 4, respectively).

No resistance could be measured below the critical current, and at the switching current a direct transition to the normal state occurs. Thus the $I_c(\Phi/\Phi_0)$ measurements really probe the purely superconducting state (although being at the frontier with the normal state). This in contrast with the classical $R(\Phi/\Phi_0)$ measurements involving the presence of thermally activated phase slips.[16] The results presented here are thus insensitive to heating effects. Note also that when the magnetic field is varied, the loop is kept in the normal state by applying a large current but the same results are obtained when the magnetic field is varied under no applied current. Since the geometry and the current bias are quite symmetric, the current flows equally in the left and right arms of the loop. Figure 2(a) depicts schematically what would have been the critical current evolution of a perfect loop if each arms had the same critical current (I_{c0}). When the flux is a multiple of Φ_0, the circulating current is zero so the total critical current is maximum and amounts to $2I_{c0}$. As soon as the flux deviates from those values, the critical current alternates (every $\Phi_0/2$) between a switching induced in the left or right side of the loop (actually in the arm where the measuring current adds with the circulating one). The situation is thus periodically repeated, involving a different quantum number for the fluxoid. As it can be seen, the critical current we measure presents also a clear saw-tooth behavior where most of the points sit on a set of parallel segments with positive or negative slope depending on the current direction. The period of 1.1 mT leading to one flux quantum through the ring implies its outer area $S=1.82$ μm^2 (contrary to Ref. 13 but here our relatively large width allows to better discriminate between the inner and the outer area). The observed critical current is far below the depairing current and it logically corresponds to the depinning of field-induced vortex. Indeed, the depairing velocity $v_d=\Delta/(\hbar k_F) \sim 200$ m/s and the number of Cooper pair $n_{CP} = m/(\mu_0 \lambda^2 e^2)$ (where λ is the magnetic field penetration depth) give a depairing current density $j_d = n_{CP} q_{CP} v_d = 0.31$ A/μm^2, i.e., five times larger than what we observe here. Since the switching current is determined here by the vortex pinning strength, it can differ from one segment to the other. In our case, the pinning strength of the left arm is smaller than the one of the right arm. Under symmetrical bias the critical current is thus always reached first in the left arm. At say 4.5 mT, the flux threading the loop is just a multiple of Φ_0 so $I_p=0$ and the same critical current is found whatever the current direction. Between 4.5 and 5 mT, $I_p > 0$ and directed opposite to the measuring current if $I>0$ (4 to 1) leading to an increase of the critical current, while if $I<0$ (1 to 4) I_p adds and the critical current is decreased. Between 3.9 and 4.5 mT, $I_p<0$ and exactly the opposite addition or subtraction is observed between I_p and I. At 3.9 and 5 mT, the flux differs from a multiple of Φ_0 by $\Phi_0/2$ so I_p is maximal. If the vortex pinning strength would have been equal in the two arms leading to the same critical current, the minimum of the set of data curves in Fig. 2(b)

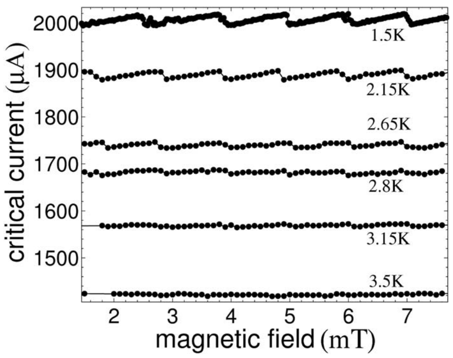

FIG. 3. Critical current vs magnetic field of a Nb loop measured with symmetric leads ($I>0$) at various temperature. The height of the saw-tooth modulation, which amounts to 23 μA at 1.5 K, is progressively reduced to 18, 11, 8, and 5 μA as the temperature is raised to 2.15, 2.65, 2.8, and 3.15 K respectively.

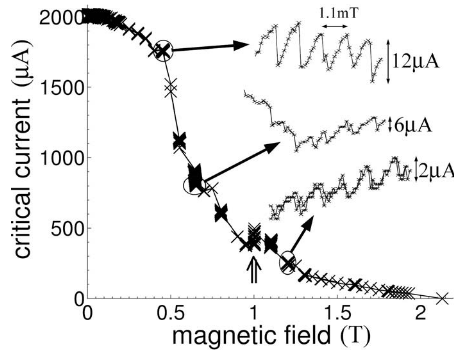

FIG. 4. Critical current vs (positive) magnetic field of a Nb loop measured with symmetric leads at 1.5 K. Current flows from leads 4 to 1. The three insets magnify the saw-tooth behavior observed around 0.45, 0.65, and 1.2 T with 0.1 mT steps (measured up and down around 1.2 T). The double arrow indicates a small matching effect.

would have been observed. We also note that in general, the total flux in the loop is

$$\Phi = \Phi_{ext} + \Phi_I + \Phi_{I_p} \approx \Phi_{ext} \tag{2}$$

where Φ_{ext} is the external flux, Φ_I cancels since left and right arms carry the same current and have similar inductances, and $\Phi_{I_p}=LI_p$, where L is the loop inductance, is the flux created by the circulating current. Here $L=1.7$ pH, so even when I_p is maximal it produces only 1% Φ_0 and has thus a negligible effect on the periodicity.

The temperature evolution of the modulation of the critical current with the magnetic field is shown in Fig. 3. At 1.5 K, the saw-tooth height amounts to 23 μA (about 1% I_c) and corresponds to $I_{pmax}=11.5$ μA. As the temperature rises, the saw-tooth modulation is still observable up to 3.15 K with roughly always the same periodicity ($\sim$1.1 mT) but the height of this modulation decreases drastically and amounts already only to 5 μA at 3.15 K ($0.3\% I_c$) before becoming almost unnoticeable at 3.5 K. The fact that I_p seems to decrease faster than the critical current itself may indicate that thermal fluctuations help the vortex motion and lead to a rounding of the saw-tooth modulation.

The critical current has also been recorded versus magnetic field up to high field (Fig. 4). It presents a strong peak effect[17] in the 0–1.25 T range and then drop to zero when the vortex are free to move at the irreversibility field $H_{irr}=2.13$ T. This peak effect results from the random (strong) pinning site lattice of this material. Some coincidental matching may occur between this natural pinning lattice and the vortex lattice leading to small local maxima of the critical current as observed at 1 T. The height of the critical current saw-tooth behavior drastically reduces as the field is increased (when vortices start to be less strongly pinned on the sites) but it is remarkable that at say 1.2 T where several hundreds of vortex are present in the loop, the impact of the circulating current can still be observed. Each vortex is indeed surrounded by a flow of superconducting current, but any of these currents give a contribution to the circulating current because of the cancellation due to their revolution symmetry. Only the circulating current flowing around the loop and in between vortices gives rise to a measurable contribution of the oscillating behavior.

We now consider an asymmetric biasing of the loop by using the contacts 1 and 2 (of Fig. 1) as presented in Fig. 5.

The approximation of Eq. (2) remains valid. Indeed, since the two loop arms have the same section S, their respective inductance ratio L_1/L_2 is equal to their length ratio l_1/l_2. However, the ratio of the current flowing in each arms is $I_1/I_2=(l_1/l_2)^{-1}$ since

$$I_{1,2} = \frac{\Phi_0}{2\pi} \frac{1}{L_{k_{1,2}}} \Delta\theta \tag{3}$$

where $\Delta\theta$ is the phase difference between the two contacts and $L_{k_{1,2}}$ the kinetic inductance of each branch given by $L_{k_{1,2}}=\mu_0\lambda^2(l_{1,2}/S)$. Therefore, $\Phi_I=L_1I_1-L_2I_2$ is strictly zero

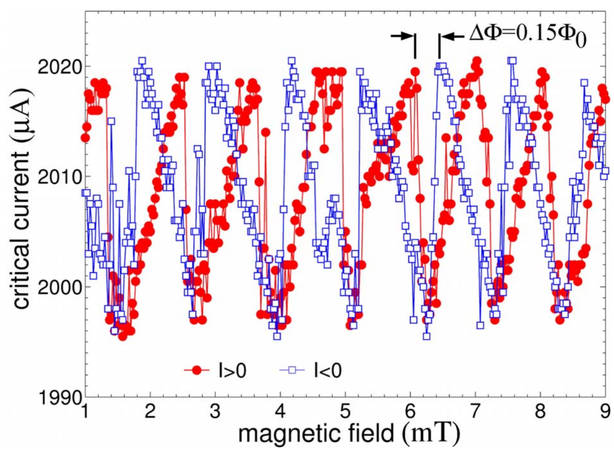

FIG. 5. (Color online) Critical current vs magnetic field of a Nb loop measured at 1.5 K with asymmetric leads. For each value of the (positive) magnetic field, the critical current is subsequently measured in the positive (red dot) and negative (blue square) current direction (current flowing from leads 1 to 2 and then 2 to 1, respectively).

in our case. We note that since here $l_2=5l_1$ and according to Eq. (3), there is 5 times more current flowing between contacts 1 and 2 in the short arm (length l_1) than in the long arm (length l_2). Critical current in arm l_1 is thus larger than in the left side of the loop (1675 μA vs 1004 μA deduced from the symmetrical case) simply because differences in the vortex pinning strength. Since about 83% of the current flows in the short arm between contacts 1–2, switching occurs there under this asymmetric bias. Also, as Fig. 5 shows, the $I_{c+}(\Phi/\Phi_0)$ and $I_{c-}(\Phi/\Phi_0)$ dependences remain periodic in asymmetric ring but the position of their extrema shifts slightly by $\Delta\Phi=(0.15\pm0.05)\Phi_0$. Introducing an asymmetry by varying the width $(w_{1,2})$ rather than the length $(l_{1,2})$ of the loop arms, the authors of Ref. 13 reported anomalous shift reaching a maximum value $\Delta\Phi=0.5\Phi_0$ as soon as the asymmetry level w_1/w_2 exceeds 1.25. This is in apparent contradiction with quantization condition 1 and with $R(\Phi/\Phi_0)$ measurements. Here, despite our very large asymmetry level, $l_1/l_2=5$, only a very small shift is observed. We attribute this to the fact that in our case, $\Phi_I=0$. The small shift that only remains can probably be attributed to the flux created by the current leads in the asymmetric case ($\Delta\Phi=0$ in the symmetric case because then the leads couple no flux).

Regarding the magnitude of the circulating current I_p, we can recall that its aims is to bring the total flux threading the loop to the nearest number of flux quantum. Thus, from the observed saw-tooth height ΔI_c, we can derive a global inductance L characterizing this loop using $\Phi_0=L\Delta I_c$. We obtain $L\sim100$ pH at 1.5 K and low field. Since the geometrical inductance is negligible, this global inductance reflects mostly the total kinetic inductance of the loop. As the temperature is increased or as the external magnetic field is increased, the magnetic field penetration depth λ becomes larger and so ΔI_c decreases, as observed experimentally. The same experiment has been done on a loop of about the same geometry but made of NbN by reactive sputtering deposition and using the same patterning technique. For this sample, no modulation of the critical current could be seen even at 1.5 K and low magnetic field. This can be understood easily since in the case of NbN, λ is 10 times larger than for Nb and therefore the kinetic inductance is 100 times larger (so I_p is 100 times smaller).

In conclusion, we have observed the presence of the circulating current in a niobium loop resulting from fluxoid quantization. Unlike precedent observations, the circulating current is here observed far from T_c and directly from $I_c(\Phi/\Phi_0)$ in a loop with a very small coherence length compared to the other loop dimensions. The presence of this circulating current could still be detected at very large field, where several hundreds of vortex are present in the loop. The flux periodicity, governed by the flux quantum, is determined by the outer area of the loop. Furthermore, when a large loop asymmetry is present in such a way that no significant flux is created by the measuring current, we found only a very small shift on the magnetic field characteristics when the direction of the current is inverted, in contradiction to what was observed before.[13] These results extend the methods for probing vortex depinning effects in superconductors.

We thank C. Ulysse and L. Couraud for the Nb deposition. This work was supported by the Interuniversity Attraction Poles Program (P6/42)-Belgian State-Belgian Science Policy. S.M. is supported by the National Fund for Scientific Research (FNRS) Belgium.

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