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Current–voltage characteristics of Pb and Sn granular superconducting nanowires

Sébastien Michotte,[a] Stefan Mátéfi-Tempfli, and Luc Piraux
Unité de Physico-Chimie et de Physique des Matériaux (PCPM), Université Catholique de Louvain, Place Croix du Sud 1, B-1348 Louvain-la-Neuve, Belgium and Research Center on Microscopic and Nanoscopic Electronic Devices and Materials (CERMIN), Université Catholique de Louvain, B-1348 Louvain-La-Neuve, Belgium

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Arrays of granular superconducting Pb and Sn nanowires (40–55 nm in diameter and 22 or 50 μm long) have been prepared by electrodeposition in nanoporous membranes. A simple technique has been developed to perform electrical transport measurement on a single nanowire. By sweeping the dc current inside the nanowire, we observed the formation of phase-slip-centers far below the critical temperature. In contrast, in voltage-driven experiments, an interesting S-shaped behavior has been observed in the nucleation region of these phase-slip-centers. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1582356]

It has been known for a few decades that a critical current I_c only perturbs, not suppresses, superconductivity in a sample of low dimensionality. Instead of depinning the vortex array (as for a type-II bulk superconductor) and finally turning the sample normal, this current induces a nonequilibrium phenomenon and leads to the successive nucleation of phase-slip-centers (PSCs) arising from Josephson-like pulsations. This behavior was first observed with conventional superconductors in the form of whiskers[1,2] or microbridges,[3] but it is also present, for example, in thin films of conventional superconductors or high-temperature superconductors,[4] and thus it always generates a lot of interest. The simplest geometry to study these PSCs is a one-dimensional (1D) superconductor having a diameter smaller than the coherence length, which is thus too narrow to accommodate a single vortex and where these PSCs can be viewed as the substitute to the vortex flow process. However, the previous studies on whiskers and microbridges in this 1D regime did not allow observation of PSCs far below T_c because the increased power dissipation produces local heating. For $T-T_c \gtrsim 0.1$ K, the PSC nucleation was hidden by fully normal spreading hot-spots,[5] even when a weak spot was deliberately introduced to depress I_c, which lowers the heat dissipation.[6] In contrast, our nanowire system presents the advantage to remain in this 1D regime even far below T_c, and the heating level is sufficiently low to allow the observation of these PSCs.

The superconducting nanowires were prepared by electrodeposition into the nanopores of homemade track-etched polycarbonate membranes.[7] For the lead nanowires, a 22-μm-thick membrane (with pore diameter ~40 nm and pore density $\sim 4\times 10^9$ cm^{-2}) and an aqueous solution of 40.4 g/l $Pb(BF_4)_2$, 33.6 g/l HBF_4, and 15 g/l H_3BO_3 were used.[8,9] In the case of tin nanowires, a 50-μm-thick membrane (with pore diameter ~55 nm and pore density $\sim 2\times 10^9$ cm^{-2}) and an electrolyte of 41.8 g/l $Sn(BF_4)_2$ in water solution were used. A constant potential of -0.5 V versus an Ag/AgCl reference electrode was used in a three-electrode configuration in order to reduce the Pb^{2+} or Sn^{2+} ions into the pores. As can be seen on nanowires prepared from the same membrane and under the same conditions (Fig. 1), the nanowires are cylindrical and the diameter is uniform along their length.

In order to perform electrical transport measurements on a single nanowire, a self-contacting technique has been developed [Fig. 2(a)]. In addition to the thick gold cathode (~1 μm) from which the nanowires start to grow up inside the membrane, another thin gold layer (in the range of 50 to 200 nm) was deposited on the other side exposed to the electrolyte prior to electrodeposition. This contacting layer covers the surface, but, contrary to the cathode, it is not thick enough to plug the pores. As for these small pore diameters the nanowires do not grow at the same speed, the first emerging nanowire interrupts the growth of the others by favoring the formation of a film of the deposited material on this contacting layer. This electrodeposited film also presents the

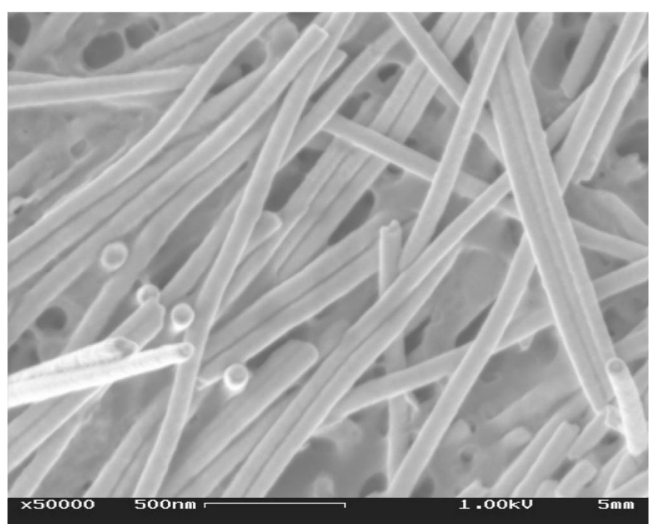

FIG. 1. Scanning electron microscopy-field emission gun (SEM-FEG) micrograph of lead nanowires after dissolution of the hosting polycarbonate membrane with dichloromethane.

[a]Electronic mail: michotte@pcpm.ucl.ac.be

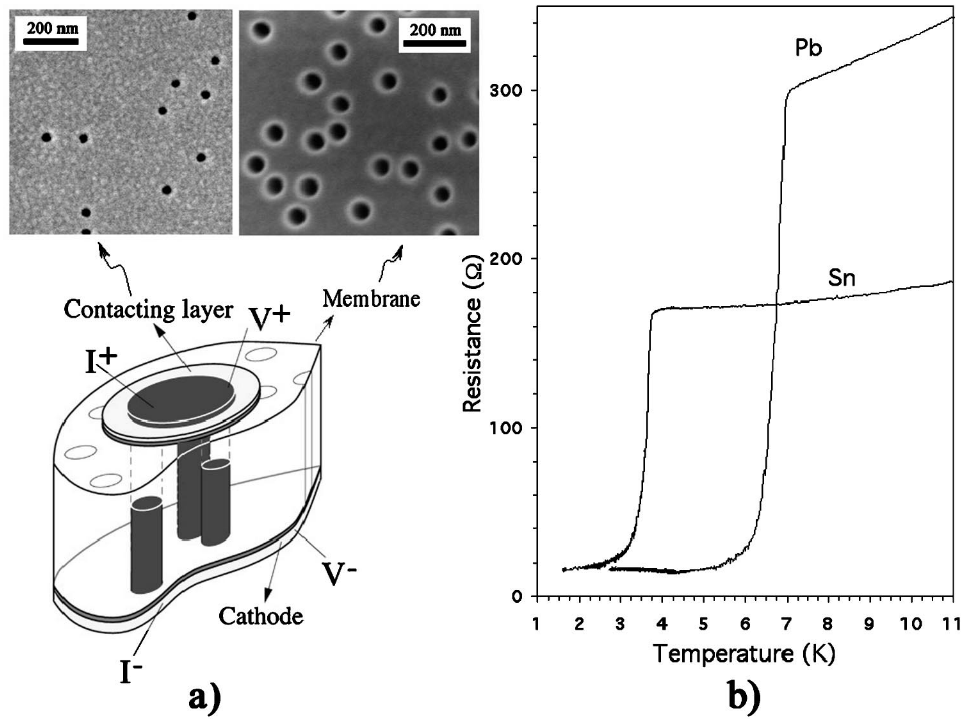

FIG. 2. (a) Schematic drawing of the sample illustrating the self-contacting technique and SEM-FEG micrographs of the membrane and the gold contacting layer. (b) $R(T)$ of a Pb nanowire (40 nm in diameter, 22 μm long) and of a Sn nanowire (55 nm in diameter, 50 μm long) measured with a lock-in amplifier by applying a small current of 0.1 μA.

advantage of perfect encapsulation of the nanowire inside the membrane, insuring protection against oxidation.

All the transport measurements were done in an electromagnetically shielded environment. Our cryostat was adequately grounded and filtered. Low-noise triax connectors and cables were used as well as Keithley sources and voltmeters. The low-temperature resistance of a 40-nm Pb nanowire (22 μm long) and a 55-nm Sn nanowire (50 μm long) is shown in Fig. 2(b). The critical temperature is not far from its bulk value: 7.2 K for lead and 3.7 K for tin. From the numerous measurements performed both on Pb and on Sn nanowires, it appears that only small depressions of T_c were observed. In addition, a superconducting transition was found to occur when the residual resistivity ratio RRR is larger than about 2, while for smaller RRR values, an insulating behavior is observed. Therefore, our nanowires are in the granular limit and moreover are moderately disordered: in the present study, the RRR of the samples was larger than 10. Structural characterization of the Pb nanowires using transmission-electron-microscopy (TEM) experiments confirms also this granular character.[8] In this context, the T-independent resistance far below T_c could be a signature of the metallic part of the superconductor–metal–insulator (S–M–I) transition[10] although the contribution from the contact resistance can hardly be subtracted. Moreover, in granular superconductors, disorder favors quantum fluctuations of the phase of the order parameter, rather than suppression of the amplitude of this order parameter. This is why only small depressions of T_c were observed.

In Fig. 3, the dc voltage–current characteristics of the Pb and Sn nanowires are reported, applying either a dc current or a dc voltage. In the two types of experiments, no hysteresis was observed. These measurements were also performed with reversed polarity, without any change in the results. In the current-driven experiment, which is the one usually performed, we observe for the Pb nanowire in Fig. 3(a), the successive appearance of two PSCs at low temperature (e.g., at $I = 50$ and 63 μA, respectively, at $T = 4.3$ K). According to the phenomenological Skocpol–Beasley–Tinkham (SBT) model,[3] on time average, 65% of total current is supercon-

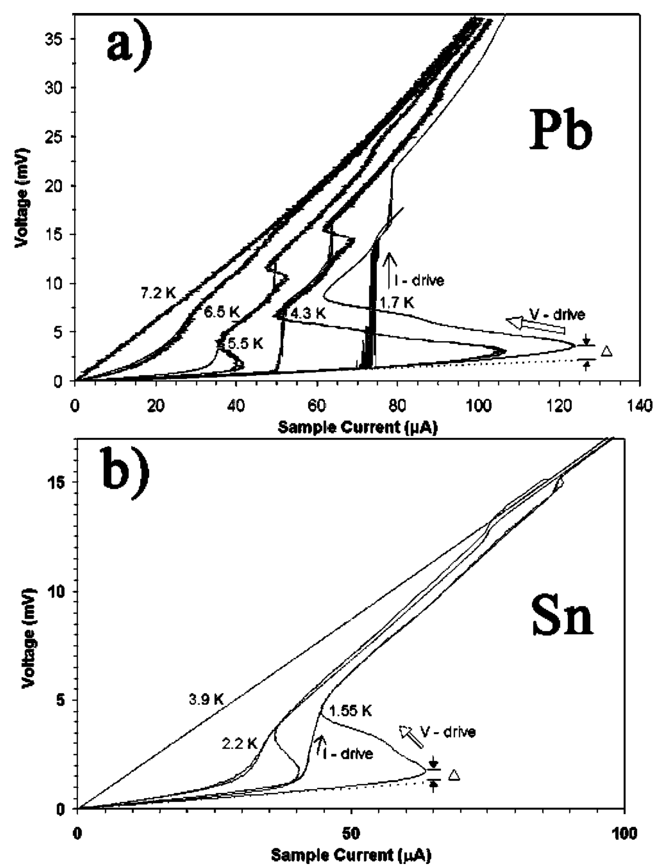

FIG. 3. Voltage–current characteristics at different temperatures of (a) a Pb nanowire (40 nm in diameter, 22 μm long) and (b) a Sn nanowire (55 nm in diameter, 50 μm long). Open arrow, the dc voltage measured versus the applied dc current (I-drive). Filled arrow, the dc current through the nanowire versus the applied dc voltage (V-drive).

ducting inside a PSC. The size of the first PSC can then be estimated from the ratio of the first voltage jump to the total corresponding normal voltage for a 22-μm-long nanowire. The value thus estimated (18 μm) is reasonable because it is twice the quasiparticle's diffusion length, which is typically of the order of 10 μm in such a superconductor. Despite the fact that successive PSCs tend to avoid those already in place, the second PSC created in this Pb nanowire is thus forced to interpenetrate the first one, which explains why the second jump in resistance is smaller. The current-driven experiment on the 50-μm-long Sn nanowire in Fig. 3(b) also shows the formation a PSC at low temperature, but for this particular material, the PSC extension is larger (around 40 μm) and the formation of a second PSC almost coincides with the transition to the normal state.

Interesting features were observed when measuring in the opposite way, that is, applying the voltage and measuring the current. Here, the current flowing into the nanowire was determined by measuring the voltage across a 1-Ω resistance added in series, and the voltage developed across the sample was measured separately. In this voltage-driven experiment, an interesting S-shaped behavior occurs at low temperature in the formation region of these PSCs, both for the Pb and the Sn nanowires [see Figs. 3(a) and 3(b)]. Although there have been some theoretical predictions showing such S-shaped behavior,[11] a detailed understanding of this behavior is still lacking. Therefore, we are restricted to pointing out some interesting experimental observations, at the lowest measured temperatures (i.e., at 1.7 and 4.3 K for Pb and 1.55 K for Sn), where the nature of the contact (normal in this case) becomes unimportant due to the Andreev reflection process.[12] At the first maximum in current, the voltage obtained by subtracting the linear $I-V$ part due to the residual resistance from the measured one (see Fig. 3) corresponds closely to the value of the gap Δ ($\approx$1.35 mV for Pb and $\approx$0.56 mV for Sn). This tends to indicate that in this voltage-driven experiment, Cooper pairs are sped up until they reach the depairing velocity $\mathrm{v}_d=\Delta/(\hbar k_F)$. The nanowire then reached progressively, through the negative differential resistance branch of the curve, the state corresponding to the PSC-like one in the current-driven experiment.

It should be noted that heating effects are unavoidable in such experiments. Indeed, the heat produced by Joule heating in the PSC has to be evacuated mostly through the media surrounding the measured nanowire. Although the thermal conductivity of polycarbonate is rather low, the surrounding media is, in fact, a composite material consisting not only of the polymer medium, but also of metal nanowires, due to the high density of nanopores [see Fig. 2(a)]. These other nanowires are not contacted electrically to the contacting layer, but they also contribute to heat transfer in the composite material due to their large thermal conductivity. The bending of the curve seen for the largest currents of Fig. 3(a) is a signature of heating effects in the measurements of the Pb nanowire. It should, however, be emphasized that this effect is almost negligible in the experiment involving the Sn nanowire [Fig. 3(b)], likely due to a smaller contact resistance and/or better heat evacuation. As a consequence, although the exact shape of the reported S-behavior for the Pb nanowire is probably slightly modified due to the temperature increase, the heating effects do not throw the S-shaped behavior into question.

In conclusion, we have developed a simple method to electrically contact single nanowires such as Pb and Sn. This technique allows us to probe the nonequilibrium properties of such 1D superconductors and in particular to observe the formation of PSCs at temperatures much below the critical temperature. Moreover, we observed an unusual behavior when a voltage is applied to such nanowires, which still requires theoretical explanation.

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