The fabrication of reproducible superconducting scanning tunneling microscope tips

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Superconducting scanning tunneling microscope tips have been fabricated with a high degree of reproducibility. The fabrication process relies on sequential deposition of superconducting Pb and a proximity-coupled Ag capping layer onto a Pt/Ir tip. The tips were characterized by tunneling into both normal-metal and superconducting films. The simplicity of the fabrication process, along with the stability and reproducibility of the tips, clear the way for tunneling studies with a well-characterized, scannable superconducting electrode.

I. INTRODUCTION

Scanning tunneling microscopy (STM) has been proven to be an important tool in the study of superconducting materials by serving as a local probe of the density of states (DOS) near the Fermi energy. STM studies of superconducting materials are conventionally performed in the S/I/N (superconductor/insulator/normal-metal) configuration, where a sharp normal-metal tip is brought to within tunneling distance from a superconducting sample. A natural and intriguing extension of the capabilities of STM studies would be to use a superconducting tip, thus allowing for S/I/S measurements.

Although superconducting tips have been used in the past in an attempt to form S-S point contacts, true vacuum tunneling has not been achieved until quite recently. The difficulty in fabricating superconducting tips arises mainly from the conflicting needs of making an atomically sharp tip, while keeping the tip apex clean and well defined. Since most known superconducting materials readily oxidize, the fabrication of a superconducting tip is significantly more complicated than that of a normal-metal tip, where metals like Pt and Au can be used. In addition, one may be concerned that the tunneling current through a very small junction area may lead to current densities that are sufficient to destroy the superconducting state at the tip. This issue was addressed by Meservey who argued that currents, typical of normal STM operation, could be carried without excessive depairing.

Pan et al. have demonstrated that quasiparticle tunneling from a sharp superconducting tip is indeed possible. They have used tips cut from Nb wire and cleaned in-situ by field emission. Although the work of Pan et al. provided an important step towards the implementation of superconducting tips in STM studies, their method of tip fabrication did not yield sufficiently reproducible results, and a large variation in the superconducting properties of their tips was observed. It may be argued that the field emission cleaning procedure used did not remove all of the Nb oxides from the tip. There are a variety of oxides in Nb, out of which NbO₂ and Nb₂O₅ are insulators and their removal can be verified by successful tunneling at low temperatures. However, NbO₂ is metallic at 4.2 K and any residue of this material on the tip apex, which is not directly detectable, may result in a proximity layer of unknown thickness. To our knowledge, no method for the fabrication of reproducible superconducting tips has been reported yet.

In this article we demonstrate a new, simple method for the fabrication of superconducting tips. By use of a controlled Pb/Ag proximity bilayer deposited onto pre-cut Pt/Ir tips, we have obtained superconducting tips with a high degree of reproducibility. Furthermore, due to the slow oxidation rate of Ag and the mechanical rigidity of Pt, our tips can be easily manipulated ex-situ without any significant degradation of their superconducting properties.

II. FABRICATION AND CHARACTERIZATION

Pt₀.₈/Ir₀.₂ tips were cut from a 0.25 mm diameter wire. We verified the quality of these tips by imaging the surface of a highly ordered pyrolytic graphite single crystal at room temperature. The tips with the best resolution were selected, and placed in a bell-jar evaporator such that the tip axis was pointing in the directions of the evaporation sources to prevent shadowing effects. A 5000 Å thick layer of Pb was deposited on the Pt/Ir tips by thermal evaporation at a rate of ~50 Å/sec. Without breaking vacuum, a thin layer of Ag was subsequently evaporated at a rate of ~2 Å/sec. The Ag layer thickness was varied from 30-60 Å in four different deposition runs. The pressure during the two-step evaporation process was ~4×10⁻⁶ torr or lower.

Pb (Tc=7.2 K) was chosen so that at 4.2 K the tips would be well below their transition temperature and the layer thickness was chosen to assure bulk superconductivity in the Pb layer (ξ₀=830 Å). The silver serves as a capping layer, protecting the lead from rapid oxidation upon exposure to the atmosphere. The thin Ag layer is proximity-coupled to the lead layer, and it has been

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FIG. 1. Temperature dependence of the superconducting gap for a tip with $d_{Ag}=30$ Å. Inset- normalized conductance curves at various temperatures.

FIG. 2. Conductance curve at 2.0 K for the same tip as in Fig. 1. The lead transverse (T) and longitudinal (L) phonon features are clearly observed, their position ($V_T-\Delta=4.5$ meV and $V_L-\Delta=8.5$ meV) and magnitude consistent with literature (Ref. 13). Inset- schematic depiction of the experimental setup.

...demonstrated\cite{footnote} that the Pb/Ag proximity junction is reproducible and stable. The Pb-Ag combination is also a good metallurgical choice, as there is no significant alloying at the interface.\cite{footnote}
The tip is therefore expected to behave as an effective superconductor with $T_c$ and $\Delta$ only slightly below that of bulk lead.

We have found that our tips can be stored in air for a period of about two days before an oxide layer is formed, which is thick enough to prohibit tunneling at low temperatures. The lifetime of the tips can be appreciably extended when stored in rough vacuum, and we have successfully used tips after three weeks of storage under these conditions.

In order to characterize the tips, we have performed tunneling measurements on a Au sample in the temperature range 2.0-6.3 K, using a custom built ultra high vacuum low temperature STM.\cite{footnote} Ultra high purity $^4$He exchange gas was used to thermally couple the STM to a liquid He bath, whose temperature could be lowered to 2.0 K by lowering the He vapor pressure. Measurements above 4.2 K were performed while the STM was allowed to slowly warm up. Differential conductance was measured using standard lock-in techniques. At each bias voltage sweep, 512 data points were taken at a rate of 3.0 ms per data point. Data was typically averaged over 250 (100) ramps below (above) 4.2 K. The junction resistance was 100 MΩ with the bias voltage during the feedback cycle fixed at +50 mV with respect to the grounded sample. Since the Au sample is a normal metal with a constant density of states (DOS) near the Fermi energy, the obtained curves represent the tip DOS.

Typical results are presented in Figure 1. Conductance curves (shown in inset) were fitted to BCS theory using the Dynes, Narayanamurti, and Garno DOS\cite{footnote} allowing both $\Delta$ and $\Gamma$ to vary, while the temperature was fixed to the value read by a thermometer mounted on the sample holder. We believe these values accurately reflect the...
perconducting tips and a Pb film deposited in-situ on a graphite substrate. These spectra are characteristic of an S/I/S junction, and conductance peaks (A) are clearly visible at the expected voltages corresponding to \( eV = \pm (\Delta_{\text{tip}} + \Delta_{\text{Pb}}) \). The peak corresponding to the gap difference (B) disappears as expected, as the temperature is lowered and the number of available states due to thermal excitations across the gap is reduced. At higher temperatures this peak is located at zero bias, which confirms again that the superconducting gap value of the tip is very close to that of lead.

It is remarkable that the tip structure is conserved despite the relatively large amount of material deposited onto it. Since our STM is optimized for large area scans, and atomic resolution images are therefore not ordinarily obtainable, we can only give an estimate on the lower bound of the tip resolution. This was done by imaging the profile of step edges on graphite, and taking the apparent width of the step to be characteristic of our instrument resolution. Using this procedure we obtain resolutions of 5-20 Å. Since the growth of Pb is granular in nature it is reasonable to assume that the tip resolution is limited by the size of the Pb grain at the apex of the tip. Limiting the mobility of Pb atoms during the deposition, by using higher deposition rates, using a Ge seed layer, or by cooling down the tip during deposition, would decrease the Pb grain size and probably improve the tip resolution.

III. CONCLUSION

In summary, we have demonstrated a new method for the fabrication of superconducting tips. We have shown that this method, relying on proximity coupling between a Pb superconductor and Ag capping layer, is capable of yielding reproducible, clean, and stable tips with \( T_c \) and \( \Delta \) close to that of bulk Pb. The observed tunneling characteristics in both S/I/N and S/I/S configurations and their temperature dependence are consistent with expectations.

Many possible applications emerge from the availability of well characterized, reproducible superconducting tips, ranging from thermometry to spin polarized tunneling. Recently there has been increased theoretical interest in the use of superconducting tips for tunneling experiments into high-\( T_c \) superconductors, where information regarding the order parameter symmetry can be obtained by tunneling near a grain boundary in such materials.

Arguably, one of the most intriguing applications is the possibility of forming a scannable Josephson junction. Because of the inherently high resistance of a vacuum tunnel junction, the Josephson coupling energy, \( E_J \sim \hbar \Delta / e^2 R_{NN} \), where \( R_{NN} \) is the junction normal resistance, is expected to be smaller than or comparable to the thermal energy in the system. In this case, strong phase fluctuations will have a dominant effect on pair tunneling, as was shown by Ivanchenko and Zil’berman. Preliminary studies have shown that low temperature I-V curves for junction resistances around 50-100 kΩ are in agreement with the theory of Ivanchenko et al.

To conclude, our newly developed tips may open the way to a variety of STM studies previously out of reach, most notable is the possibility for a scanning Josephson tunneling microscope.

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