Novel Methods of Fabrication and Metrology of Superconducting NanoStructures

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***Abstract*—As metrology extends toward the nanoscale, a number of potential applications and new challenges arise. By combining photolithography with focused ion beam and/or electron beam methods, superconducting quantum interference devices (SQUIDs) with loop dimensions down to 200 nm and superconducting bridge dimensions of the order 80 nm have been produced. These SQUIDs have a range of potential applications. As an illustration, we describe a method for characterizing the effective area and the magnetic penetration depth of a structured superconducting thin film in the extreme limit, where the superconducting penetration depth λ is much greater than the film thickness and is comparable with the lateral dimensions of the device.**

***Index Terms*—Magnetic field effects, nanotechnology, superconducting devices, thin films.**

I. Introduction

THE DEMANDS of metrology move toward ever more sensitive measurements on ever-smaller systems driven by industrial and scientific requirements. The dimensional metrology of thin-film structures with lateral dimensions in the range of 100 nm to 100 μm and thicknesses in the range of 1–100 nm is a topic of increasing interest. Traditionally, superconducting devices such as superconducting quantum interference devices (SQUIDs) have been used at the macroscopic scale, but increasingly, there are potential applications at the nanoscale. These nanodevices can be used for a range of applications, including single-particle detection, spintronics, or quantum information processing. With the addition of appropriately configured antennas, applications in photon detection and spectroscopy at millimeter wavelengths or frequencies in the THz range become possible. In this paper, we describe how focused-ion-beam (FIB) milling and/or electron-beam lithography can be combined with conventional photolithography to produce nonhysteretic SQUID devices having loop dimensions of the order 200 nm and superconducting bridge dimensions as small as 80 nm. We describe the fabrication and characterization of these devices and demonstrate how they may be incorporated into measurement devices. We point out that for applications in nanoscale, single-layer thin-film SQUIDs are better suited than conventional devices incorporating trilayer Josephson junctions. Due to their intrinsically higher critical current densities and lower specific capacitances, microbridges permit the minimizing of both junction inductance and capacitance, which are essential to optimize SQUID energy sensitivity at the nanoscale [1]. To ensure that microbridges are close to ideal Josephson devices, it is necessary to deal with issues of adequate thermal shunting (through bridge geometry) and avoidance of hysteresis (through shunt-resistor fabrication) [2]. These issues will be outlined in Section II.

SQUIDs offer unique measurement capabilities for a wide range of physical parameters, from displacement to current or voltage. In this paper, we demonstrate the utility of nanoscale SQUIDs with reference to a specific example of the measurement of the effective superconducting penetration depth in a thin-film structure. Apart from practical difficulties encountered in properly defining their physical boundaries, the penetration of electromagnetic fields into superconducting structures presents a set of unavoidable additional problems for metrology applications. For example, complications arise since the penetration length λ will vary strongly with temperature, frequency, and film thickness. We have developed a noninvasive *in situ* method to measure the temperature-dependent magnetic properties of ultrathin superconducting structures. We have employed this technique for characterizing effective inductance and penetration depth in the extreme limit, where λ is much greater than the film thickness or even its lateral dimension. This method is applied to our devices, and we show that numerical and quasi-analytical methods show results that are in good agreement with the experiment.

II. Film Deposition, Device Fabrication, and Testing

We first emphasize that the magnetic properties of thin films and devices made from them are extremely sensitive to the deposition conditions and thickness of the films. Accurate dimensional control is possible only when the films themselves have been characterized, and as the relevant dimensions approach the submicrometer or nanometer scale, an *in situ* method

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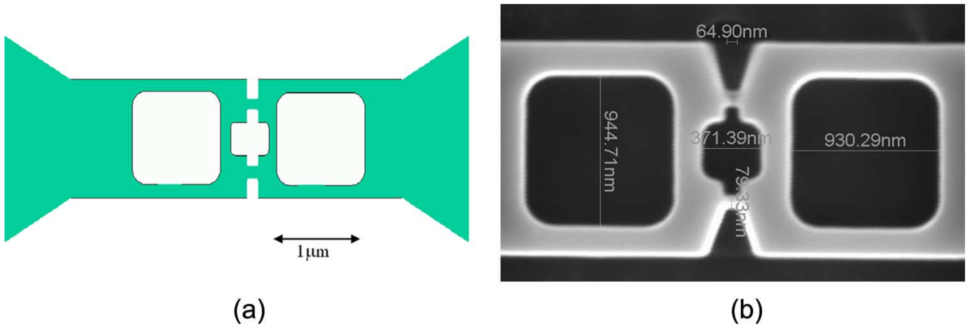

Fig. 1. (a) Schematic design of prototype superconducting structure with submicrometer dimensions. (b) SEM of actual device prepared from a Nb thin film by FIB milling.

is required to establish, for example, the magnetic penetration depth of the films used in a particular type of device. In this section, we therefore focus on the details of the film materials and the fabrication technique we have developed for preparing devices, which incorporate nanometer-scale features.

Crucially, our current application of FIB techniques has allowed the traditional photolithographic fabrication route to be retained and extended into the submicrometer and nanometer scale. Niobium is chosen as the basis of our low-temperature superconducting devices because of its long-established use, conveniently adjustable transition temperature T_c (4.2–9.4 K) [3], and the availability of suitable fabrication and clean-room facilities. Niobium films are deposited by dc magnetron sputtering from a 99.999% pure target onto oxidized silicon wafers or other suitable substrates (e.g., MgO and Al_2O_3) held near room temperature in an ultrahigh-vacuum chamber with a base pressure around 10^{-7} Pa. By controllably varying the film thickness from 14 to 200 nm, a range of T_c from 5 to 9 K is achieved for the purposes of our experiment [4], [5].

For the SQUID devices used in this paper, Josephson junctions of microbridge type are used. In addition to the advantages previously noted, this design simplifies the fabrication process, compared with the traditional trilayer technique. The critical submicrometer bridge regions are individually prepared by FIB milling of the appropriate regions after the overall device structures such as the one shown in Fig. 1(a) have been delineated by photolithography and reactive-ion etching. A shunt resistor is formed by a 150-nm layer of tungsten hexacarbonyl, $W(CO)_6$, which is deposited over the Nb tracks by using an electron beam. This layer prevents Ga ion implantation in the Nb junction during milling and subsequently acts as a suitable resistive shunt. During milling, simultaneous imaging of the sample with the electron beam is possible, allowing the milling to be stopped as soon as all the desired Nb is removed. To illustrate the capabilities of the technique, Fig. 1(a) shows a schematic design of a prototype structure incorporating submicrometer dimensions, whereas Fig. 1(b) shows a scanning electron micrograph (SEM) image of the completed device produced by FIB milling of a Nb thin film. A close-up of a bridge region is shown in Fig. 2. The superconducting characteristics of typical nanobridges made in this way are shown in Fig. 3. The critical current is about 240 μA at a temperature of 8.75 K. Note that there is no sign of hysteresis, and the microbridge junctions show near-ideal resistively shunted junction behavior.

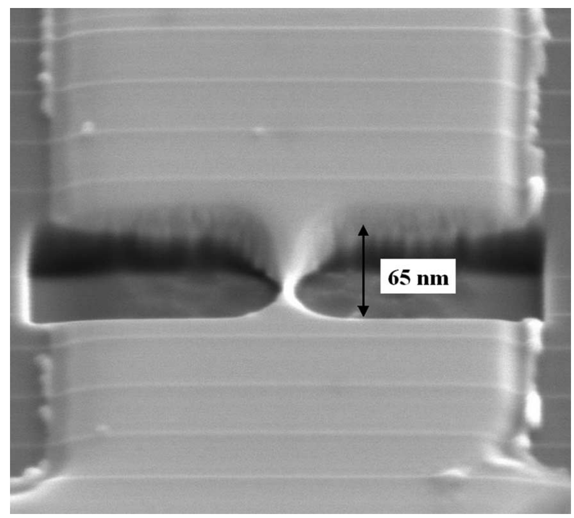

Fig. 2. Close-up SEM image of superconducting Nb nanobridge structure ~80 nm wide produced by FIB milling.

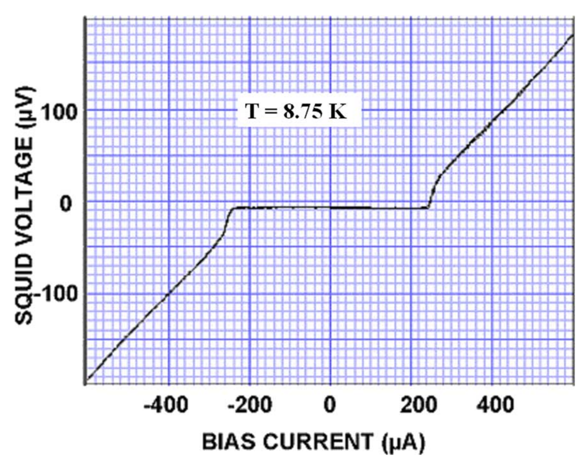

Fig. 3. I–V characteristic for a SQUID containing two nanobridge junctions of the type shown in Fig. 2.

III. Application of NanoSQUIDs to NonInvasive Measurement of Penetration Depth

Measurements of the penetration lengths in thin superconducting films have usually been based on the two-coil method or variations thereof [6]–[8], where the lateral dimensions of the film are necessarily on the order of 10 mm due to the dimensions of practical wire-wound coils. Other techniques [9] are restricted to the high-frequency (GHz) range. With a reduction of the dimensions of superconducting devices into the submicrometer regime (e.g., for quantum computing or single-particle counting applications), we have developed [10] a noninvasive *in situ* technique for measurement of the temperature-dependent magnetic properties of ultrathin superconducting structures. The method allows measurement of the effective area and penetration length in ultrathin-film structures of micrometer dimensions and can be extended to more general measurements of magnetic properties of nanoscale structures. It is based on the conventional definition of a SQUID's effective area $A_{\rm eff}$, which is described as follows:

$$A_{\rm eff} = \Phi_0/\Delta B \tag{1}$$

where Φ_0 is the magnetic flux quantum, and ΔB is the change in applied field required to produce a response of one period in the SQUID's voltage versus field output. The technique is inherently broadband (dc to MHz) in frequency.

We consider one of the simplest situations of interest: a plane lamina of radius a and thickness d, which is exposed to a perpendicular dc magnetic field B. The structure has its superconducting normal transition at $T = T_{c1}$. The London penetration depth λ is such that $d < \lambda < a$. The 2-D (Pearl) length $\Lambda = \lambda^2/d$ is then comparable to or greater than the lateral dimension of the patch a in the operating temperature range $0.5T_{c1} < T < T_{c1}$. To simulate the application of a nanoscale SQUID making measurements on the thin-film structure, we note that the lamina is contained within a superconducting SQUID ring, which has a transition temperature $T_{c2} \gg T_{c1}$. Over the temperature range of interest, the ring remains fully superconducting. The calculations/modeling can be modified or extended to determine how the magnetic properties of the disk are affected by the presence of the ring.

For $T < T_{c1}$, the effective area $A_{\rm eff}$ and penetration length Λ are related by the following equation (for a circular disk in this example, but other geometries can be similarly calculated):

$$A_{\rm eff} = \pi(a + 2\Lambda(T))^2. \tag{2}$$

Rewriting this, we derive the effective penetration length Λ and its temperature dependence from measurements of $A_{\rm eff}$ as follows:

$$\Lambda(T) = 0.5((1/\pi){A_{\rm eff}}^{0.5} - a). \tag{3}$$

The SQUID–disk combination resembles a conventional SQUID at temperatures $T_{c1} < T < T_{c2}$, whereas at temperatures in the interval $0.5T_{c1} < T < T_{c1}$, the effective area will increase with temperature up to some limit at which the disk (but not the surrounding SQUID ring) is entirely in a nonsuperconducting state.

In our experiments, the devices have been cooled in a well-shielded enclosure to liquid helium temperatures on a stage that can be temperature controlled in the range of 1.4–15 K or alternatively to as low as 100 mK by using an adiabatic demagnetization refrigerator. The temperature stability is better than 0.005 K. A 50-turn field coil produces a magnetic field up to 50 μT at the SQUID. Four terminal dc connections enable conventional I–V and V–Φ characteristics to be measured by using a room-temperature preamplifier. For more sensitive measurement, an ac bias current at 49 kHz is applied to the SQUID, and a low-temperature tuned transformer provides optimum coupling to the amplifier. In a typical experiment, a steadily increasing magnetic field is applied perpendicular to the plane of the SQUID, such that the SQUID's sinusoidal voltage-field characteristic undergoes an exact number of periods, as shown in Fig. 4. From repeated measurements, the mean value of ΔB corresponding to one flux period enables a mean value of $A_{\rm eff}(T)$ to be evaluated from (1). By repeating the procedure at a range of temperatures, the corresponding temperature dependence of the penetration length $\Lambda(T)$ for the ultrathin superconducting patch is evaluated from (3). The flux response for two different temperatures is shown in Fig. 4. For comparison, identical SQUIDs that were fabricated without the internal superconducting patch showed no temperature-dependent change in effective area over the same temperature range. Note the large change in period between the two curves, demonstrating the strong contribution of the temperature-dependent Pearl effective penetration depth to the effective area of the SQUID loop. The effective area can increase by a factor of two as the temperature increases from $0.7T_{c1}$ to $0.95T_{c1}$. A typical set of our data, obtained by measurements of the SQUID's effective area, is compared with results of microwave transmission experiments [9] in Fig. 5. We note that the measured values of penetration length, ranging from 100 nm to 10 μm, can easily exceed the geometrical dimensions of typical structures such as those of Figs. 1 and 2. The present method allows us to make these measurements on thin-film samples just a few micrometers across, whereas the techniques employed by Wang *et al.* [3] and Gubin *et al.* [9], in common with others, required samples at least 10 mm in diameter.

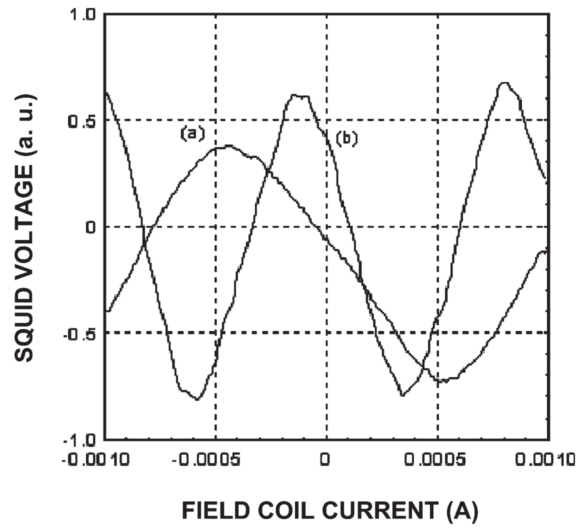

Fig. 4. Voltage response of a SQUID with an enclosed superconducting disk (see text) to an applied magnetic field (proportional to solenoid field current) at two different temperatures (a) $T = 4.5$ K and (b) $T = 6.2$ K.

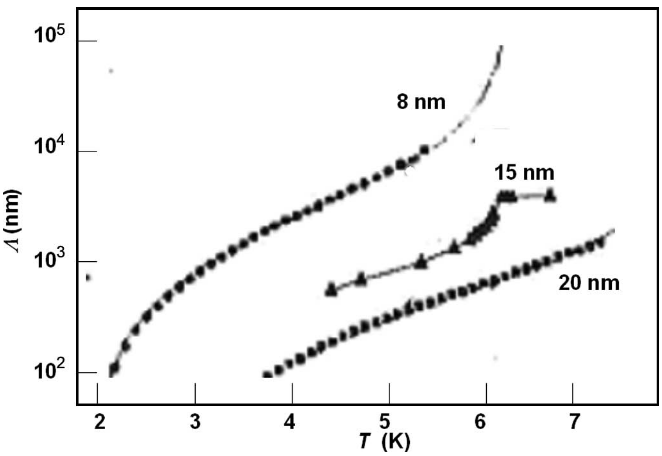

Fig. 5. Comparison of measured values of the temperature dependence of penetration length in Nb films (15 nm thickness, present technique) with results of a microwave technique (8 nm and 20 nm thickness, Gubin *et al.*, [9]).

IV. Discussion and Conclusion

We aimed to show in this paper that SQUID technology has much to offer to the new science of nanoscale metrology. In order to address metrological needs at this important new length scale, it is essential first to solve a number of new fabrication problems. We have shown that the combination of electron-beam lithography or FIB milling with conventional photolithography can produce nanoscale microbridge weak links that have near-ideal Josephson properties. This procedure represents a much simpler and more appropriate solution to the problem of reduced physical scale than conventional trilayer processes can offer.

To illustrate the application of these nanoscale microbridge SQUIDs, we have demonstrated that the temperature dependence of the penetration depth of a thin superconducting film may be measured noninvasively, and that the technique covers a greater range both of physical dimensions and frequency bandwidth, compared with previous methods. The devices are robust, readily prepared, and characterized so that a wide range of metrological applications may be expected to follow.

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