SQUID techniques. I. Obtaining reliability in point-contact SQUID's


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(Received 4 April 1974; in final form 30 May 1974)

Stable and permanent niobium point-contact SQUID's have been fabricated by using oxidized niobium screws to form the point contact. The reliability of these devices is further ensured by a simple and versatile environmental shielding procedure. Over a period of five years there have been no failures of such SQUID's in our laboratory. The signal-to-noise performance of these SQUID's is quite good.

The preparation and properties of reliable permanently adjusted rf-biased point-contact SQUID's are discussed in this paper. We are reporting our procedures since they apparently yield devices superior to those generally available. The essential features of our SQUID's are the oxidized niobium screws which form the point contact and the unique but simple enclosure which provides protection from environmental hazards. Complete reliability in combination with the excellent signal-to-noise performance makes these point-contact SQUID's highly competitive with present thin-film devices.

During the past several years, advances in the technology of superconducting quantum flux detectors (SQUID's) have resulted in a rapid growth of SQUID applications. Various types of quantum flux detectors have been developed, but the most common is the rf-biased SQUID which operates with transitions between adjacent fluxoid quantum states. These devices consist of a superconducting ring closed by a single Josephson-like weak link. The ring is inductively coupled to an rf tank circuit which is typically driven at a resonant frequency of \( \sim 10 \text{ MHz} \), although considerably higher frequencies have been used.

There are basically two competitive versions of the rf-biased SQUID which are distinguished by the method of forming the superconducting weak link. One is the thin-film SQUID where a constriction in the film constitutes the weak link. The other version is the bulk niobium SQUID where a permanently adjusted point contact acts as the weak link.

The chief attributes of the thin-film SQUID are its ruggedness and reliability. Condensation and other normal environmental hazards seldom have any effect upon the device. Due to the low normal-state resistance \( R_n \lesssim 0.5 \Omega \) of present thin-film weak links, they are resistant to damage by stray electrical discharges, but they usually suffer from relatively high noise and often from high sensitivity to operating temperature fluctuations.

The major unfavorable characteristic of present thin-film SQUID's appears to be substantially inferior signal-to-noise performance compared to point-contact devices. Unfortunately it is difficult to discuss this quantitatively since signal-to-noise data are seldom transmitted except through verbal communications. There is a lack of published detailed numerical discussions of SQUID performance, particularly at very low frequencies. This seems to be especially the case for the thin-film SQUID's. However it does appear that the best reported noise performance has been obtained with point-contact devices rather than thin-film SQUID's, both for the standard \( \sim 10 \text{ MHz} \) SQUID and the new higher-frequency devices. The origin of this apparent excess noise in thin-film SQUID's is uncertain. Experiments in our laboratory have indicated two possible sources: (i) a nonsinusoidal current phase relation in the thin-film weak link due to its relatively large dimensions, and (ii) flux motion in the thin-film ring.

Point-contact SQUID's generally have excellent signal-to-noise performance and reasonable operating temperature insensitivity. However, the available devices often suffer from a very serious defect—lack of reliability. In developing the prototype for most contemporary rf point-contact SQUID's, Zimmermann, Thiene, and Harding were able to obtain a permanently adjusted thermally stable device, yet others with similar point-contact SQUID's have experienced fairly frequent device failures. These failures usually occur as drastic changes in the point-contact critical current that are variously attributed to electrostatic discharges, corrosion by water condensation, or deformation due to thermal stress. The frequent point-contact readjustments that are required as a consequence of these changes is a severe price to pay for the signal-to-noise advantage that point-contact SQUID's offer. We proceed to describe our procedures for avoiding this problem.

The basic design of our point-contact SQUID's is shown in Fig. 1. It is quite similar to the symmetric two-hole niobium block design developed by Zimmerman et al. The device is fabricated by drilling two 0.109-in. holes in the niobium block and then spark cutting the 0.010-in. slot connecting the holes. Two opposing 000-120 screw holes are drilled and tapped in the center of the block perpendicular to the slot. As shown, niobium lock nuts are used to maintain the niobium screws in place once the point contact has been made.

The key difference between our device and other point-contact SQUID's lies in the preparation of the point contact. We use the usual combination of one mechanically sharpened and one flat 000-120 niobium screw, but the flat screw is lightly oxidized before the room-temperature point-contact adjustment is made. This oxidation is accomplished simply by heating the screw in air on a hot plate for a few minutes at \( \sim 150^\circ \text{ C} \) until a light gold
FIG. 1. A schematic drawing of the niobium point-contact SQUID. The flat niobium screw is lightly oxidized before the point contact is made.

The oxidizing technique usually produces a point contact that is an S-\(S'\)-S junction, whereas unoxidized point contacts are more often S-\(S'\)-S' junctions.\(^{13}\) (S represents the bulk niobium, \(S'\) a complicated mixture of insulating and superconducting oxides, and \(S'\) a thin bridge of niobium.) We have found that the temperature dependence of the critical current \(I_c\) of oxidized point contacts is much stronger than that of point contacts formed between clean niobium metal. To illustrate this we give some data from measurements of this temperature dependence for the two different cases in Fig. 2. The data show that for clean contacts \(I_c\) exhibits a rather weak temperature dependence not unlike that predicted by Aslamazov and Larkin for superconducting metallic point contacts.\(^{14}\) On the other hand, \(I_c\) of oxidized contacts almost invariably changes approximately exponentially with temperature. This result indicates that the oxidized point-contact junctions are not simple tunnel oxide junctions but, perhaps surprisingly, are more like the normal metal junctions of Clarke.\(^{15}\)

Because of this stronger temperature dependence, SQUID's using oxidized point contacts must operate within a smaller temperature range. However, such SQUID's seldom have an operating range of less than 1 K, and they can readily be adjusted to operate at any desired point in a wide temperature interval. In our laboratory SQUID's operating at temperatures as low as 0.5 K and others at temperatures as high as 7 K have been routinely produced.

Perhaps unexpectedly it is only in oxidized point-contact SQUID's that the ideal predicted\(^{16,17}\) intrinsic noise limits have been obtained.\(^{11,18}\) Measurements in our laboratory have also revealed that the superconducting current phase relation of an oxidized point contact is almost exactly the simple Josephson sinusoidal relation.\(^{19}\)

Although a niobium oxide layer can be generated by electrochemically anodizing the niobium screw, we have found that this is not a satisfactory substitute for heating the niobium screw in air. Scanning electron micrographs have shown that our thermal oxide layer is quite flaky and irregular, while an anodic oxide layer of comparable thickness is very tight and uniform. We have found that it is rather difficult to achieve a satisfactory superconducting contact through even a relatively thin anodic

FIG. 2. Some representative data of the temperature dependence of the critical current of (a) clean niobium point contacts and (b) oxidized niobium point contacts. \(R_N\) is the normal-state resistance of the point contact.

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layer, while it is very easy to do so through the thermally generated oxide. Whenever suitable anodized point contacts have been achieved, their stability, particularly with respect to temperature cycling, has been entirely unsatisfactory. Apparently a more "perfect" oxide layer is less desirable than a rather imperfect coating.

The ease of adjustment and the excellent nature of the weak link are decided advantages of oxidized point contacts, but their most important features, in comparison with unoxidized point contacts, is their superior stability and reliability. We can speculate on the reasons for this difference. The mechanical nature of the oxide layer may figure in this effect since the rough oxide apparently acts as a support protecting the contact area from damage by mechanical shocks. Indeed we have found that these oxidized point contacts are rather highly resistant to mechanical shocks. We have repeatedly dropped permanently adjusted oxidized point SQUID's which were encapsulated as discussed below from a height of over 3 ft to a tile floor, with no detectable change in the critical current of the device.

The most dominant factor in the enhanced reliability is almost certainly the electrical characteristics of oxidized point contacts. The normal-state resistance at 4.2 K of an oxidized point contact which has been adjusted to the correct critical current, ~5 μA, for satisfactory SQUID operation is typically of the order of 1 Ω. The normal-state resistance of an unoxidized point contact similarly adjusted ranges from 10 to 100 Ω. Thus, oxidized point contacts have a considerably higher "fusing" level than unoxidized contacts and can tolerate a greater current flow before suffering damage. This lower susceptibility to electrical discharge damage is a major factor in the enhanced reliability of oxidized point-contact SQUID's.

We have found that for a given oxidized point contact the resistance of the contact is essentially temperature independent, typically varying by no more than 20% from room temperature to 4.2 K. Thus, if one can measure by some means the room-temperature resistance of the oxidized point contact of a SQUID while making the adjustment, a resistance in the range 1–1.5 Ω, will result in a critical current at 4.2 K of ~5–10 μA. This is provided, of course, that repeated adjustments are not made so many times that the oxide is worn from the niobium. Indeed, for best results in a permanently adjusted configuration, no more than two repeated contacts should be made before reoxidizing the niobium.

We have also developed a simple encapsulating procedure that provides necessary environmental shielding of the device. It consists of hermetically sealing the permanently adjusted SQUID into a superconducting shielded can. As shown in Fig. 3, the SQUID is clamped in a micarta holder and the tank circuit is inserted into one of the holes of the SQUID by the proper amount to obtain optimum coupling. This assembly is placed into a lead-plated OFHC copper can which has a reentrant 0.010-in. wall brass tube protruding inside so as to fill the second hole of the SQUID. As shown, the top of this tube is sealed while the bottom opens to the outside through the bottom of the can. This permits insertion of a flux transformer by which a signal can be coupled into the SQUID. The tank circuit (the capacitor is not shown) is connected to a hermetic coaxial connector at the top of the can. The can is sealed with either a lead or indium O-ring, evacuated and filled with an atmosphere of helium gas through the pinch-off tube which is then pressure-welded shut. Once the SQUID has been sealed into this can, it is almost completely protected from the hazards of the outside environment. It can be cycled rapidly from room temperature to 4 K and back with no detrimental effects. If it is to be operated at higher temperatures or fields than the lead can will permit, a slightly more complicated sealed system may be used which consists of an inner niobium shield and an outer unplated hermetically sealed copper can.

A special feature of our shielding arrangement is the brass tube. The tube inductively shields the flux transformer from the rf drive field of the SQUID, avoiding possible negative effects the rf might have upon sensitive samples. Conversely, the brass tube shields the SQUID from rf noise and electrical discharges which the flux transformer might pick up. The cutoff frequency of the tube, 100 kHz or greater, is chosen so that its magnetic noise spectrum is sufficiently broad that it does not add significantly to the low-frequency flux detector noise. The topological arrangement resulting from the use of the brass tube also greatly facilitates the changing of the flux transformer secondary, avoiding the necessity of opening the can.

An extra benefit of the use of the brass tube is that it decreases the effective rf inductance of the SQUID by as much as a factor of 2 or more. As a result, its use significantly reduces the total noise of the SQUID detector system, affecting both the intrinsic SQUID noise and the noise component arising from dissipation in the rf tank circuit.11

![Fig. 3 A schematic drawing of the hermetically sealed can used to protect the SQUID from environmental hazards.](image-url)
The noise performance of the oxidized point-contact SQUID's is quite good. The intrinsic noise of the device itself is close to ideal, while a practical amplifier-limited rms noise level of $2 \times 10^{-4} \phi_0 / \text{Hz}^{1/2}$ is obtained while operating at 3 MHz. The long-term dc flux drift is also less than $2 \times 10^{-4} \phi_0$. Improvements in this practical noise performance should be readily attainable. Our measurements were made using an amplifier with an input noise voltage at least three times larger than that provided by the best available rf amplifiers. Further improvements can be made by operating at higher frequencies. For example, an oxidized point-contact SQUID, although of somewhat different construction and not permanently adjusted, has yielded a flux noise of $\sim 5 \times 10^{-6} \phi_0 (\text{Hz})^{1/2}$ when operated at 450 MHz.

In summary, by oxidizing the niobium screws before forming the point contact and by hermetically sealing the adjusted SQUID in a shielded can, we have been able to produce permanent highly reliable point-contact SQUID's. Devices prepared in this manner have been used routinely in a variety of low-temperature experiments in our laboratories. In five years there have been no failures of these devices, no need for any realignment of the point contact. Several such SQUID's have been operated quite regularly over this entire five-year period. The excellent noise performance of oxidized point-contact SQUID's in combination with their reliability make these SQUID's competitive with presently available thin-film SQUID's.

*Research supported by the Office of Naval Research and by the National Science Foundation through the Materials Science Center at Cornell.

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14J. E. Zimmerman, J. C. Wheatley, W. Goree, et al. (private communication).