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FABRICATION OF HIGH T_C SUPERCONDUCTOR THIN FILM DEVICES-CENTER DIRECTOR'S DISCRETIONARY FUND FINAL REPORT (PROJECT NO. P17)

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13. ABSTRACT (*Maximum 200 words*)
This report describes a technique for fabricating superconducting weak link devices with micron-sized geometries etched in laser ablated $Y_1Ba_2Cu_3O_x$ (YBCO) thin films. Careful placement of the weak link over naturally occurring grain boundaries exhibited in some YBCO thin films produces Superconducting Quantum Interference Devices (SQUIDs) operating at 77 K.

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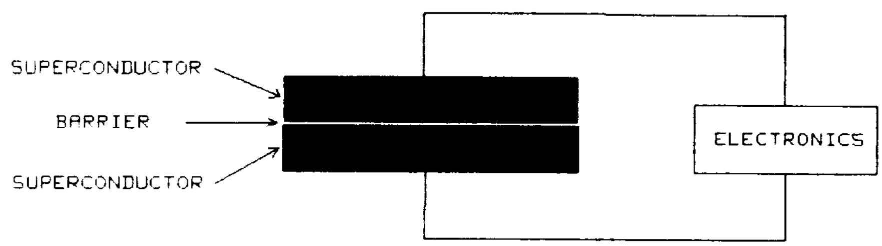
I. INTRODUCTION

Superconductivity occurs at temperatures far below anything we are likely to encounter in a normal day's activities. Low temperature superconductivity began its evolution in 1911 with the accidental discovery of zero resistance in mercury at a temperature just above 4.2 K (-452 °F). Seventy-five years later and just as exciting, superconductivity was found in a new ceramic material at temperatures high as 95 K (-289 °F). Today the material with the highest critical temperature (T_c) becomes superconducting at 125 K and newer materials hint of even higher critical temperatures in the future. Superconductors operating in this temperature range are still very cold but are significantly warmer, even "high temperature."

Superconductors are useful, depending on the application, in bulk or thin film form. Low T_c bulk materials are primarily used in the winding of superconducting magnets and must be cooled by expensive liquefied helium. Low T_c thin films are essential building blocks used in microfabricating helium-cooled radiation detectors, magnetic field sensors, and fast switches for computer applications. High T_c bulk material technology is not developed to the point of winding large coils for magnets, but high T_c thin film technology is expanding rapidly. Superconducting devices once limited to low temperature films are beginning to be fabricated in high temperature films and cooled with cheaper liquefied nitrogen.

The behavior of superconducting thin film devices is a result of the unusual behavior of the electrons in superconductors. Electrons in a non-superconducting material experience resistance to current flow and generate losses in the form of heat. Electrons in a superconductor form pairs and interact with the material's lattice in just the right way to experience no resistance, "zero resistance." In addition to this property each pair of electrons has the same wavelength and phase as any other pair of electrons. As long as the material remains superconducting it will continue to exhibit this long range order. If two separate superconductors each with like electron pair phases but a different value in each piece are brought together separated by a small distance of about 20 Å, pairs can tunnel across the barrier without any voltage. If a voltage is then applied across the barrier, the energy is radiated at a frequency proportional to the voltage. The two superconductors are no longer separate but are weakly coupled by this link and exhibit the effects predicted by Brian Josephson in 1962. A typical tunnel junction is shown in Figure 1 showing approximate thicknesses required for different barrier materials. By incorporating these barriers or Josephson junctions into closed superconducting loops and measuring the interference effects of electron waves crossing the junction, very small changes in the magnetic field can be measured. This device is called a Superconducting Quantum Interference Device (SQUID). Another type of Josephson device is capable of detecting and mixing different frequencies of microwave radiation when the junction is irradiated.



BARRIER THICKNESS: 20 ANGSTROMS FOR INSULATORS
50 ANGSTROMS FOR SEMICONDUCTORS
10000 ANGSTROMS FOR METALS

Figure 1. Josephson junction.

The approach to this project was to utilize the low T_c experience gained in the past and apply it in the new high T_c area producing SQUID devices and superconducting radiation detectors operating at 77 K. Properly designed weak links between the two superconductors replaced the very difficult to make 20 Å or less barriers used in typical tunnel junctions. The focus of this project was to fabricate micron-sized weak-link geometries from laser ablated high T_c films using optical lithography and test each device for operation.

II. MICROFABRICATION

Fabrication of micron-to submicron-wide geometries requires many carefully controlled steps. Initially the process is somewhat trial and error until critical parameters are determined. An error during any of the steps can render the device inoperable. The process relies on photolithography techniques to generate a durable master mask that is then reduced to the desired size and patterned in photoresist on the high T_c film. The resulting microbridge resist pattern is then ion-milled and tested for device operation.

A. MASK GENERATION

The relative shape of the microbridge is designed and drawn on a Hewlett Packard Model 340 Workstation running a computer-aided design program. The geometry of a typical microbridge is relatively simple and hourglass shaped as shown in Figure 2. The flexibility of a computer design allows more complicated bridge patterns to be quickly generated for additional studies as shown in Figure 3. Computer patterns can be generated in positive or negative form depending on the type photoresist used to produce the final mask. The pattern is then printed and photographically reduced to the desired size. The photograph negative is then contact-printed on a durable metal mask and anodized making the metal areas that are not protected by the photoresist transparent.

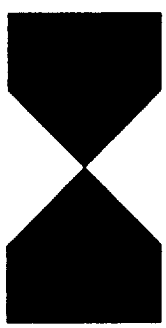


Figure 2. Single bridge.

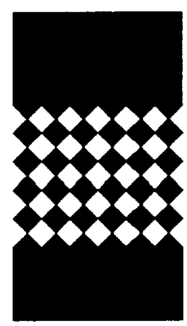


Figure 3. Multiple bridge.

B. HIGH T_c THIN FILMS

The yttrium barium copper oxide ($Y_1Ba_2Cu_3O_x$) high T_c thin films used in this project were coated by Hoi Kwok at the State University of New York at Buffalo under a previous grant. A typical superconducting film consists of a 1500 Å thick layer deposited on a magnesium oxide substrate. These laser ablated films are smooth and well suited for microfabricating micron sized geometries and have critical temperatures around 88 K.

C. PATTERN REDUCTION

A final pattern reduction is accomplished by projecting the mask image through a microscope onto the photoresist coated high T_c film. A reduced pattern of the mask is exposed in the ultraviolet sensitive photoresist coating and developed. This resulting micron-sized pattern is then ion-milled until the unprotected areas of the film are etched completely away leaving only the microbridge geometry.

The microscope system used for the final pattern reduction is a reflected light, bright field Zeiss Axioplan with additional features for imaging mask patterns on to the field. The microscope has the capability to insert, position, and center the patterned mask (metal film on glass) exactly and stably in the field diaphragm plane on a precision slider. The patterned mask is sharply imaged in the specimen plane when the sample is brought in focus. After the removal of a red filter to protect the photoresist during alignment, the reduced pattern is exposed in the photoresist. In order to obtain the sharpest focus in the ultraviolet, the microscope must be optically out of focus by approximately 1 micron. The amount of out of focus is found by a series of test exposures and once determined does not change. A schematic of this light path is shown in Figure 4 [1]. For best photoresist exposure the optics must pass wavelengths down to 350 nm and have a resolution of 0.324 μm.

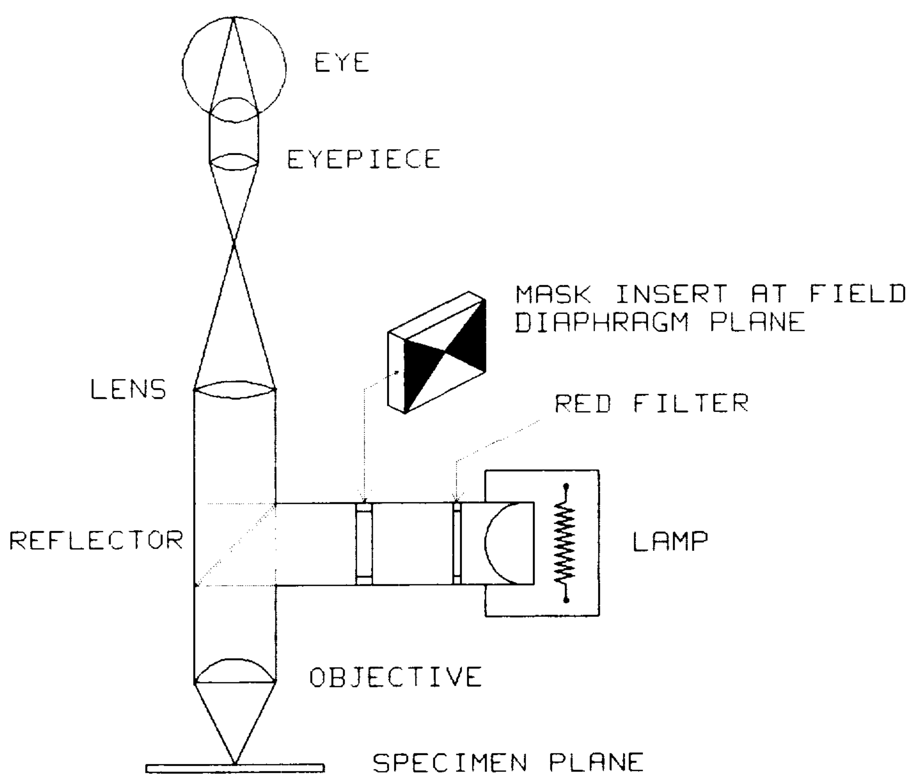


Figure 4. Light path.

D. FABRICATION PROCEDURE

The procedure for fabricating a microbridge begins with the spin coating of the high T_c film with photoresist. Shipley positive resist is applied to the surface and spun for 20 sec at 10,000 rpm. The film is allowed to dry at room temperature for a few minutes before exposing the final reduction. With the red filter in place, the microscope is focused on the film surface using the 100X objective. The patterned mask is inserted in the field plane of the microscope and optically focused. Since different frequencies of light from the microscope lamp focus at slightly different positions near the specimen, a correction must me made to insure the ultraviolet frequencies used for the exposure are in exact focus. The red filter is then removed for 30 sec exposing the resists. Shipley developer is used in a 50% mix with distilled water for 15 sec. Resulting patterns in the photoresists are examined under the microscope for proper exposure and development. A typical microbridge slightly less than 1 micron wide is produced at this stage in the process. Microbridges are produced in the high T_c film by ion milling these films for 25 min at 5 kV and 50 μA. A summary of the procedure is shown in Figure 5.

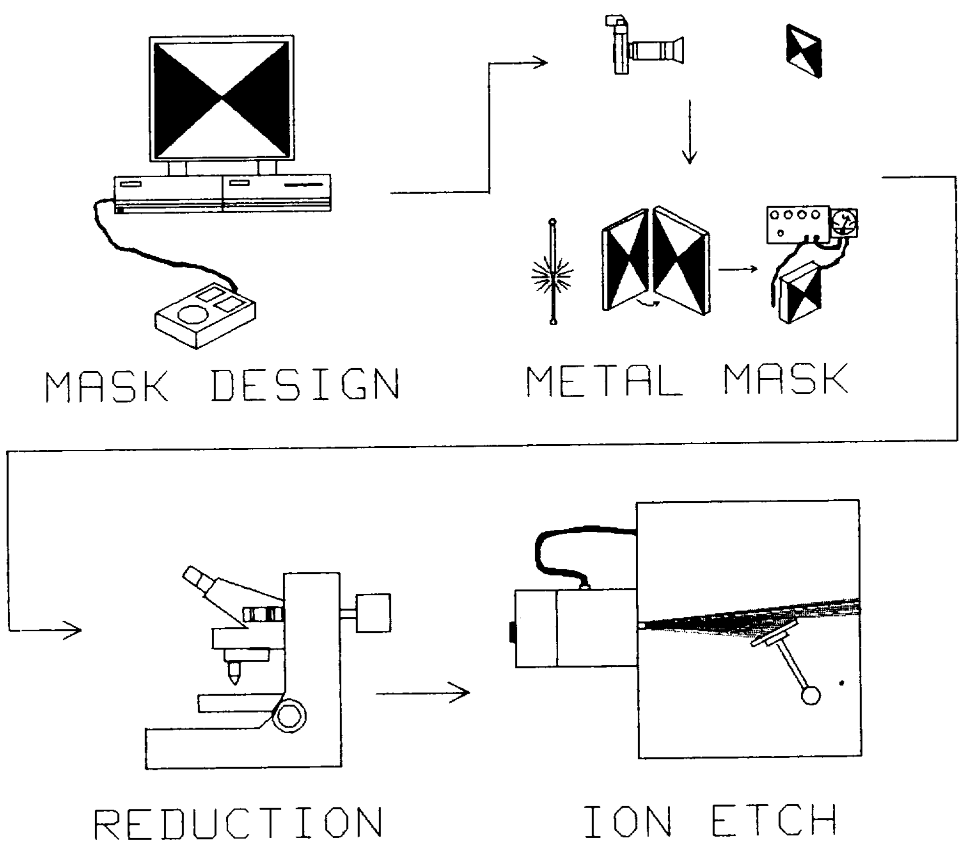


Figure 5. Fabrication process.

III. MEASUREMENTS

There are three effects associated with the long range order of pairs of electrons in a superconductor. The first effect is that the magnetic flux threading a hole in a superconductor or threading a superconducting ring exists only in multiples of a discrete quantity. This quantity is given by Planck's constant divided by two times the electron charge, since the electrons are paired, and is equal to 2.07 X 10^{-15} Weber or one flux quantum. The other two effects, predicted by Josephson, involve the barrier between superconductors. The dc Josephson effect predicts that the quantum mechanical phase of the electron pairs does not change as tunneling takes place through the barrier with no voltage applied across the barrier. If an external current (I) is passed through the barrier, the maximum value of paired electron current or supercurrent (I_c) is given by $I=I_c \sin(\phi)$, where ϕ is the electron pair phase change. The ac Josephson effect predicts that when a voltage is applied across the barrier, the energy will be radiated at a frequency given by a voltage equal to two times the electron charge divided by Plank's constant. Each microvolt placed across the barrier would give an additional increase in frequency of 483.6 MHz. In an effect related to the ac Josephson effect, steps of constant voltage during a current trace are generated when radiation falls on the barrier. The spacing between the steps will again be 483.6 MHz/μV.

One device exploiting these effects is the Superconducting Quantum Interference Device (SQUID). The radio frequency (rf) SQUID uses a superconducting loop weakened in one area by a microbridge and magnetically coupled to another loop that is part of a tuned rf tank circuit. The rf SQUID system is shown in Figure 6. A supercurrent is generated in the SQUID loop if the external magnetic field changes. This supercurrent flows in such a way as to oppose any magnetic field change. If the supercurrent generated is large enough to drive the microbridge into a voltage supporting state, individual flux quanta are free to enter or leave the SQUID loop. The rf electronics measure the voltages produced by these opposing currents which translate into external fields being detected as small as 1/10000 of a flux quantum for ideal devices.

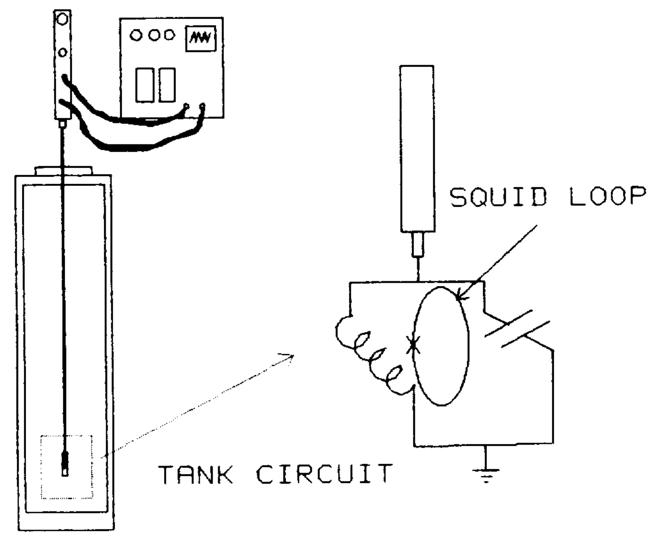


Figure 6. RF SQUID.

IV. RESULTS

The first microbridges fabricated during this project were tested for critical current (Figure 7) and critical temperature (Figure 8) using a closed cycle helium refrigerator data acquisition system [2]. The critical current value for the microbridge is approximately 0.6 mA and the critical temperature is around 80 K. Both of these values are acceptable for devices to be operated at liquid nitrogen temperatures. Many microbridges were tested but they exhibited no SQUID behavior or constant voltage steps. This was not a problem because the microbridges usually have to be further weakened. One way to weaken the structure is to fabricate the microbridge over a very fine 1 micron wide line scratched into the surface with a diamond scribe. Microbridges fabricated in this way also exhibited no SQUID behavior or constant voltage steps. This result is concerning because low temperature niobium SQUIDs have been fabricated in the past using this technique. Other attempts to weaken the microbridge area involve etching ledges a few hundred Angstroms deep into the film or etching ledges a few hundred Angstroms deep into the substrate before coating. Again none of these microbridges exhibited the desired behavior except for one microbridge. This bridge did show constant voltage steps but could not be tested for SQUID behavior because the continuous superconducting loop, necessary for SQUID operation, was carefully severed in preparation for the radiation detector measurements.

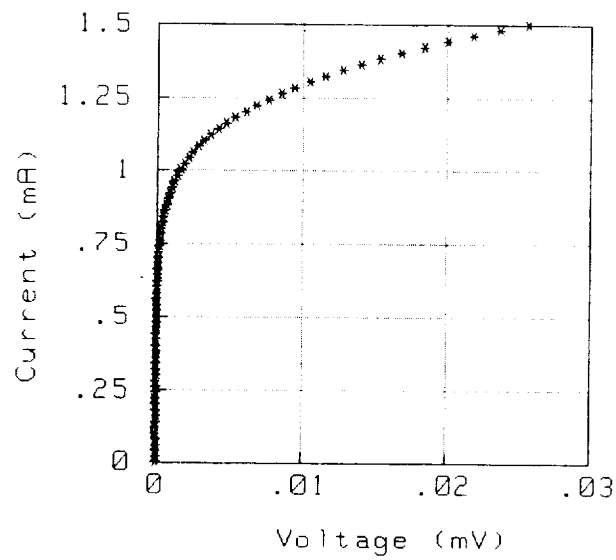


Figure 7. I-V trace.

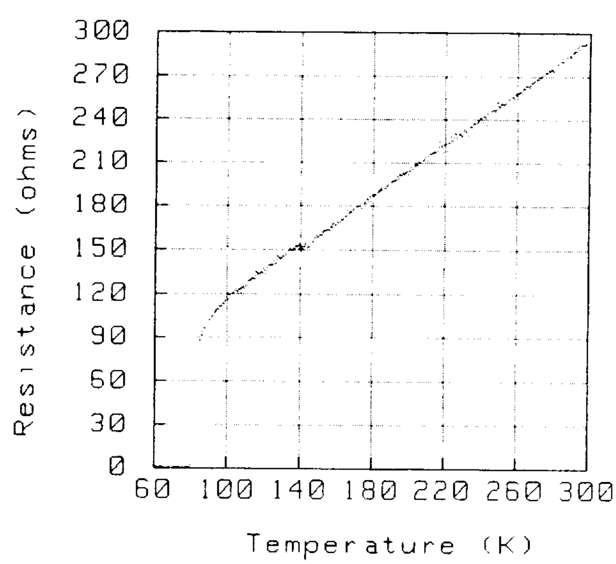


Figure 8. R-T trace.

After re-examining this working device, it was determined that the bridge was not fabricated on the ledge as intended but on some other natural feature similar in appearance to the man-made ledge. The output of this high T_c thin film device is shown in Figure 9. A current-voltage trace can be seen both without radiation falling on the bridge, the vertical line, and with several Gigahertz radiation falling on the bridge. The constant voltage steps can be seen just above the noise.

It is believed that the microbridges fabricated on scratches and ledges are still not weak enough to exhibit the desired behavior, but microbridges fabricated on what appears to be naturally occurring grain boundaries do exhibit SQUID behavior. An 800X photograph of this type of microbridge is shown in Figure 10. The difference in film texture is easily seen with an abrupt change at the junction. The SQUID output from this type of microbridge exhibits the typical periodic response to a small applied magnetic field. In Figure 11, the detected-peak-to peak change in applied field is one flux quantum.

V. CONCLUSIONS

Microbridges fabricated during this project on ledges and fine scratches showed no SQUID behavior. This result is probably related to differences in characteristics of thin films used in this effort, since others have recently reported success using this technique. Microbridges fabricated during this project on naturally occurring grain boundaries exhibit good SQUID behavior and work well at 77 K. Several devices were reproduced with similar SQUID behavior exploiting the naturally occurring grain boundaries. Very good devices can be fabricated with this technique, but man-made grain boundaries must replace the naturally occurring ones. More complicated array geometries with multiple bridges will require controlled placement and orientation of these grain boundaries. Fabricating controlled grain boundaries for producing SQUIDs was not part of this investigation, but controlled grain direction films have recently been deposited and junctions have been fabricated on controlled boundaries by others exhibiting SQUID behavior at 77 K [3].

VI. REFERENCES

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2. R. C. Sisk, P. N. Peters, NASA TM-100380, 1989.
3. L. P. Lee, K. Char, M. S. Colclough, and G Zaharchuk, Appl. Phys. Lett., 59, 23 (1991).

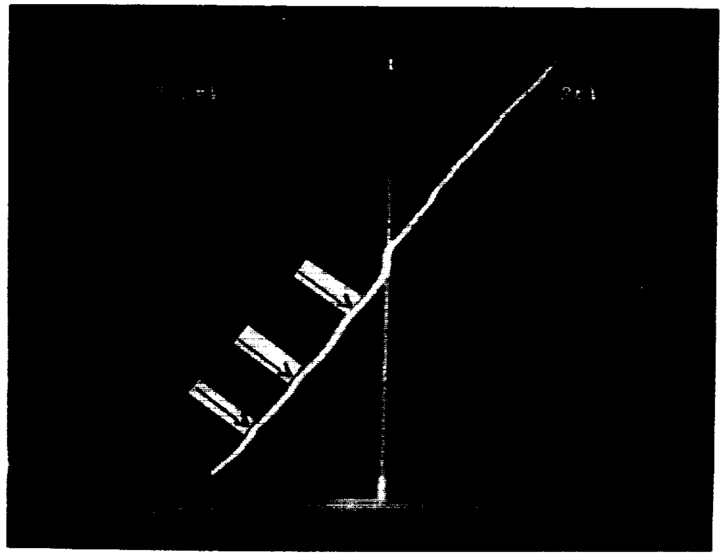

Figure 9. Constant voltage steps.

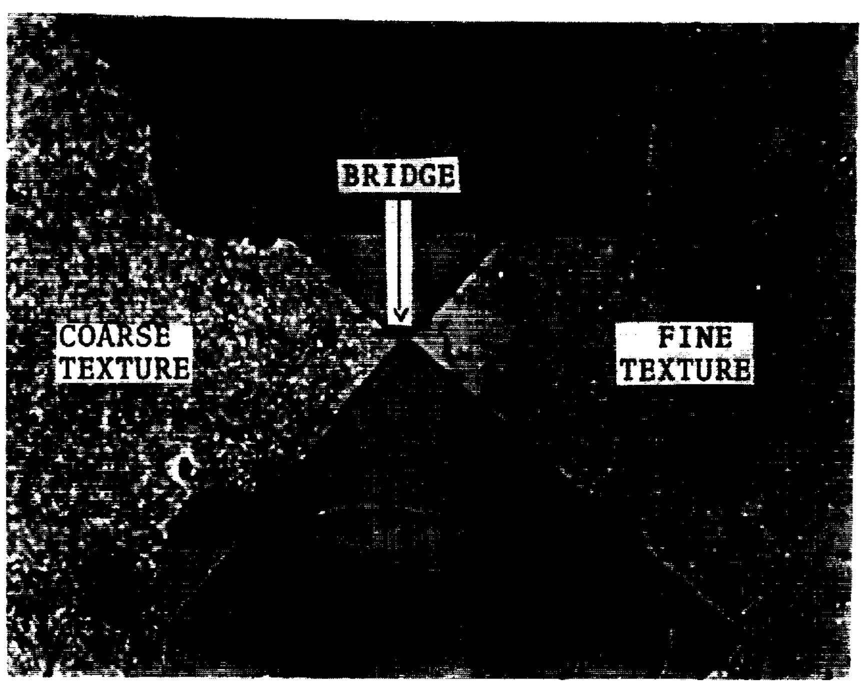

Figure 10. Microbridge (800X).

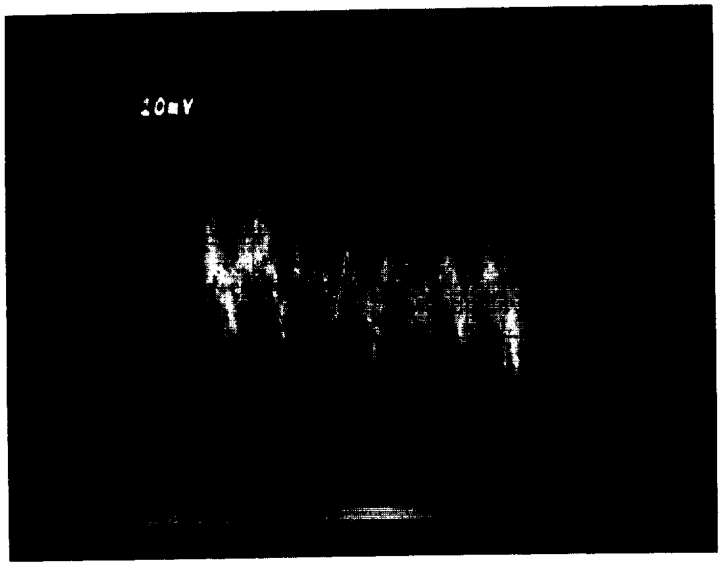

Figure 11. SQUID output.

APPROVAL

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The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

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Space Science Laboratory

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