

**Electron-Beam, X-Ray,  
and Ion-Beam Technology:  
Submicrometer Lithographies VIII**

**PROCEEDINGS**  
 **SPIE—The International Society for Optical Engineering**

# **Electron-Beam, X-Ray, and Ion-Beam Technology: Submicrometer Lithographies VIII**

**Arnold W. Yanof**  
*Chair/Editor*

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ELECTRON-BEAM, X-RAY, AND ION-BEAM TECHNOLOGY:  
SUBMICROMETER LITHOGRAPHIES VIII

Volume 1089

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ELECTRON-BEAM, X-RAY, AND ION-BEAM TECHNOLOGY:  
SUBMICROMETER LITHOGRAPHIES VIII

Volume 1089

## INTRODUCTION

The SPIE Symposium on Microlithography—and, in particular, this conference on Electron-Beam, X-Ray, and Ion-Beam Technology—is devoted to two basic principles. The first of these is practicality. This symposium has traditionally been a forum for vendors and users of advanced lithography to demonstrate materials, tools, and techniques as they are practically applied. The organizers of this conference are committed to seeing electron beams, x rays, and ion beams in actual use for making integrated circuits. The second basic principle of this symposium can be expressed by the modern aphorism, "smaller is better." Higher resolution practical lithography using nonoptical radiation is the essence of this conference and the topic of this volume.

This proceedings documents the many impressive contributions to the conference. Among the highlights are a 0.25- $\mu\text{m}$  e-beam paper with very interesting GaAs processing from Hewlett-Packard Microwave Technology Division. The Hitachi paper on hot carrier degradation occurring in MOS transistors made by e-beam lithography is a significant device paper. The use of electron cyclotron resonance plasma etching with e-beam resist is a timely contribution from Sumitomo Metal Industrial. The Fraunhofer-Institut für Mikrostrukturtechnik contributed a landmark paper on 0.5- $\mu\text{m}$  N-MOS fabricated at four lithographic levels using SOR. Automatic x-ray mask inspection using x ray itself is a very compelling and important development from Toyoda Automatic Loom Works, Aichi Institute of Technology, and Nagoya University. This list of a few of the many highlights tells more, perhaps, about the editor's personal interests than about relative quality, and the reader will certainly find his/her own particular highlights.

The conference was also greatly enhanced by the two invited speakers on x ray: Professor Heuberger from the Fraunhofer-Institut, and Dr. Kitayama from NTT Corporation. Their important papers on the state of the art are included herein.

It has been a great honor to serve once again as the chair of the SPIE electron-beam, x-ray, and ion-beam technology conference in 1989. I am very grateful to the team who selected and assembled these fine papers: cochairs Nicholas Economou, Douglas Resnick, Sheila Vaidya, and Katsumi Suzuki. Dr. Suzuki personally fields many of the high quality contributions from Japan that help make this conference significant. My great admiration also goes to the authors for the originality and hard work they put into practical accomplishments and polished presentations.

I hope to see many of you again at the SPIE conference in 1990!

**Arnold W. Yanof**  
AT&T Bell Laboratories

## INTRODUCTION

The SPIE Symposium on Microelectronics—X-Ray and Ion-Beam Technology—was devoted to two basic principles. The first of these is practicality. This symposium has traditionally been a forum for vendors and users of a particular technology to discuss materials, tools, and techniques as they are practically applied. The organizers of this conference are committed to seeing electron beams, x-rays, and ion beams in actual use for making integrated circuits. The second basic principle of this symposium can be expressed by the modern aphorism, "smaller is better." Higher resolution practical lithography using nonoptical methods is the essence of this conference and the topic of this volume.

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The conference was also greatly enhanced by the two invited speakers on a ray: Professor Hasegawa from the Fraunhofer Institute, and Dr. Kinsman from NTT Corporation. Their important papers on the state of the art are included herein.

It has been a great honor to serve once again as the chair of the SPIE electron-beam, x-ray, and ion-beam technology conference in 1989. I am very grateful to the team who selected and assembled these fine papers: co-chairs Nicholas Economou, Douglas Hensick, Sheila Vaidya, and Robert Smith. Dr. Smith personally fields many of the high quality contributions from Japan that help make this conference significant. My best admiration also goes to the authors for the originality and hard work they put into practical accomplishments and polished presentations.

I hope to see many of you again at the SPIE conference in 1990.

Arnold W. Yano  
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## SESSION 1

### ABSTRACT

### Ion-Beam Lithography

Chair

Nicholas P. Economou

Micron Corporation

### INTRODUCTION

High resolution patterning of thin film high temperature superconductors is necessary in the development of device applications of these new materials. Such fine-line structures are necessary in the fabrication of devices such as superconducting quantum interference devices (SQUIDS), transmission lines and others. Furthermore, high spatial resolution patterning can be an important tool in studying the properties of individual grains and grain boundary effects in polycrystalline thin films of the new superconductor materials.

A dc SQUID is formed with two Josephson junctions interrupting a superconducting ring [1,2]. An interference effect between the two junctions causes the critical current of the structure to be periodic with externally applied magnetic field so that in effect the device converts magnetic flux to a voltage signal. The lack of a technique for producing well defined layered structures of superconductor and dielectric films has frustrated efforts to fabricate classical superconductor-insulator-superconductor (IS) tunnel junctions with these new materials. The usual alternative of using a microbridge geometry has been tried and Josephson behavior has been observed at temperatures as high as 77K. The microbridges are generally fabricated in polycrystalline films in which the weak coupling across grain boundaries accounts for the quantum effects observed. Reproducibility and control of the critical current in grain boundary junctions is, however, poor. The main goal of our work is to develop techniques for forming planar junctions which are reliable, reproducible and have a controllable critical current. Our focused ion beam patterning has been used to fabricate a type of weak link (Anderson-Darwin) junction where a superconducting stripe is necked down to the point that the link between the banks can only support a small supercurrent because of its small cross-section. These junctions can exhibit Josephson-type behavior for sufficiently small dimensions. Our goal then was to produce these weak links with a controllable critical current (achieved by geometry) while retaining the superconducting transition onset and zero resistance temperature of the original material.

A number of techniques have been applied to patterning of high T<sub>c</sub> superconductor films, usually including resist lithography followed by wet or dry etching for pattern transfer [3-6]. Direct patterning

## Focused Ion Beam Patterning of High $T_c$ Superconductor Films

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### ABSTRACT

We have used a 20 keV Ga focussed ion beam to pattern superconducting submicron bridge structures in thin films of  $\text{Ba}_2\text{YCu}_3\text{O}_7$  material by physical sputtering. The technique can produce structures down to 0.5 microns or less in epitaxial films with no degradation in superconducting transition temperature ( $T_c$ ) or critical current density ( $J_c$ ). Photolithography was used to define a coarse pattern of 20 micron wide and 50 micron long strips, each wired for four-terminal resistance measurements. Sub-micron constrictions were then milled by the focused ion beam to form weak-link junctions with roughly 0.3 microns separating the superconducting banks. We have demonstrated that focused ion beam micromachining is capable of producing submicron sized superconducting structures and is a suitable technique for fabricating devices such as SQUIDs and the study of grain boundary effects in the films.

### INTRODUCTION

High resolution patterning of thin film high temperature superconductors is necessary in the development of device applications of these new materials. Such fine-line structures are necessary in the fabrication of devices such as superconducting quantum interference devices (SQUIDs), transmission lines and others. Furthermore, high spatial resolution patterning can be an important tool in studying the properties of individual grains and grain boundary effects in polycrystalline thin films of the new superconductor materials.

A dc SQUID is formed with two Josephson junctions interrupting a superconducting ring [1,2]. An interference effect between the two junctions causes the critical current of the structure to be periodic with externally applied magnetic field so that in effect the device converts magnetic flux to a voltage signal. The lack of a technique for producing well defined layered structures of superconductor and dielectric films has frustrated efforts to fabricate classical superconductor-insulator-superconductor (S-I-S) tunnel junctions with these new materials. The usual alternative of using a microbridge geometry has been tried and Josephson behavior has been observed at temperatures as high as 77K. The microbridges are generally fabricated in polycrystalline films in which the weak coupling across grain boundaries accounts for the quantum effects observed. Reproducibility and control of the critical current in grain boundary junctions is, however, poor. The main goal of our work is to develop techniques for forming planar junctions which are reliable, reproducible and have a controllable critical current. Our focused ion beam patterning has been used to fabricate a type of weak link (Anderson-Dayem) junction where a superconducting stripe is necked down to the point that the link between the banks can only support a small supercurrent because of its small cross-section. These junctions can exhibit Josephson-type behavior for sufficiently small dimensions. Our goal, then was to produce these weak links with a controllable critical current (scaled by geometry) while retaining the superconducting transition onset and zero resistance temperature of the original material.

A number of techniques have been applied to patterning of high  $T_c$  superconductor films usually including resist lithography followed by wet or dry etching for pattern transfer [3-6]. Direct patterning

by laser ablation has also been demonstrated [7]. These methods have been limited to feature sizes above one micron. Some of the limiting factors may have been in the nature of the film itself, chemical interactions between the process chemistry and the film, or in the case of laser ablation, thermal effects. Focussed ion beam micromachining is a direct process without process chemicals and demonstrates submicron resolution without degradation of  $T_c$  or  $J_c$ . Despite the obvious throughput limitations of such a serial process it has proven valuable in prototyping of devices which use the new superconductor materials.

## EXPERIMENTAL

The films for this work were prepared by coevaporation of  $BaF_2$ , Y and Cu in an oxygen ambient on epitaxial  $SrTiO_3$  substrates (100) and subsequently oxygen annealed. The resulting films were 2000Å thick with the C-axis up with a  $T_c$  ( $R=0$ ) near 90 K and a critical current density ( $J_c$ ) at 77 K of more than  $1 \times 10^6$  Amps per square centimeter.

The focused ion beam system has been described previously [8]. It has a single lens focusing column producing a Ga beam at 20 keV with a spot size of 0.2 micron at 160 pA beam current. The beam profile is roughly Gaussian with a sigma of  $\sim 0.085$  micron (FWHM=0.2 micron). The deflection field is 1 mm square with computer controlled stage motion of 250 x 350 mm. The system is equipped to detect both secondary electrons and mass selected secondary ions produced during image scanning or milling. The secondary ion mass spectrometry (SIMS) apparatus has been described in more detail elsewhere [9] and can provide elemental image maps, mass spectra and end point determination in multilayer structures.

The application of SIMS to end point determination when milling through  $Ba_2YCu_3O_7$  films is shown in Figs. 1-3. The mass spectrum of positive ions sputtered from the surface of the  $Ba_2YCu_3O_7$  film is shown in Fig. 1. Singly-ionized Ba appears as the most prolific secondary ion species having a counting rate of  $\sim 10^3$  cps when scanning over a  $5\mu x 5\mu$  area. Since one does not expect any substrate mass peaks to overlap (for  $SrTiO_3$ ), the presence of this signal indicates unambiguously the presence of additional film to be milled. The dose profile of the SIMS output when milling a  $5\mu x 5\mu$  square through a 2000Å thick film is shown in Fig. 2. As a supplementary check, we use the presence of a Ti signal to indicate that we have reached the substrate. For the purpose of this plot, the SIMS mass selection was multiplexed at 3 sec. intervals between the Ba and Ti. The appearance of the Ti signal at a dose of  $2.8 \times 10^{17}$  ions/cm<sup>2</sup> precedes the drop in the Ba signal at  $\sim 4 \times 10^{17}$  ions/cm<sup>2</sup>. We interpret this to indicate that the focused ion beam penetrated the film completely in some areas prior to others. This interpretation is supported by the sequence of SEM micrographs shown in Fig. 3. We see that the textured surface of the original film transfers through the milling process, and evidence of voids in the film appearing between doses of  $2 \times 10^{17}$  and  $3 \times 10^{17}$  ions/cm<sup>2</sup>. In addition, the original surface roughness may be enhanced during the milling process by any nonuniformity in the sputtering rate at grain boundaries and further amplified by the dependence of the sputtering yield on the ion beam angle of incidence.

Fabrication of the bridge structures required a photolithography step to define the superconducting banks and lead pattern (with features generally larger than 5 micron) prior to focused ion beam milling. The lead pattern used provided a set of 20 micron wide by 50 micron long strips, each wired for four-terminal resistance measurements. The sub-micron constrictions were milled by the focused ion beam in a subsequent step. The coarse lead pattern used in the experiments on annealed films were subtractively patterned on uniformly deposited films. The unwanted film was removed either by wet-etching in dilute HCl or sputter etching by 500 eV Argon ions. The photoresist mask was then removed with Acetone. Au contacts were deposited over part of the lead pattern using a tri-layer undercut photoresist /Al/photoresist stencil in a lift-off process.



2 x 10<sup>3</sup>

COUNTS

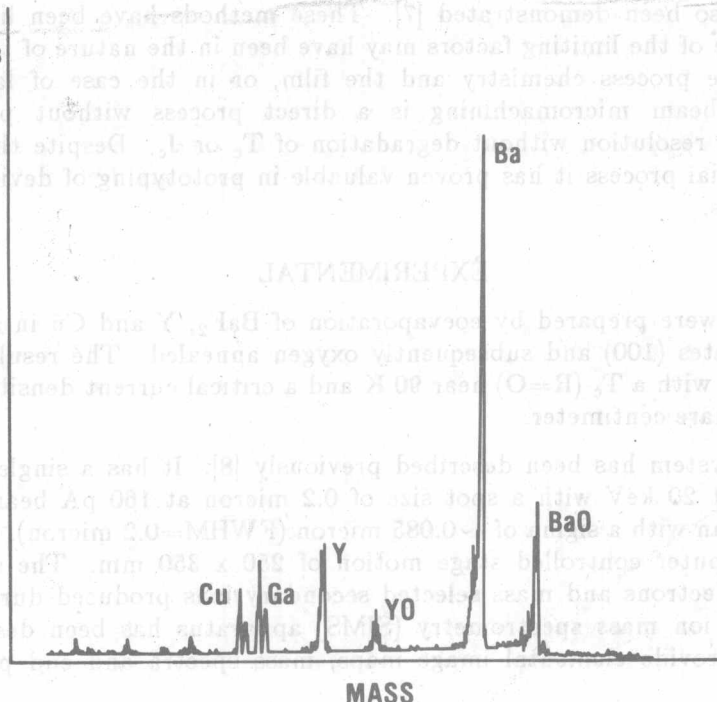


Fig. 1 SIMS mass spectrum of Ba<sub>2</sub>YCu<sub>3</sub>O<sub>7</sub> film.

10<sup>3</sup>  
COUNTS

138 Ba

48 Ti

ION DOSE x 10<sup>17</sup> IONS/cm<sup>2</sup>

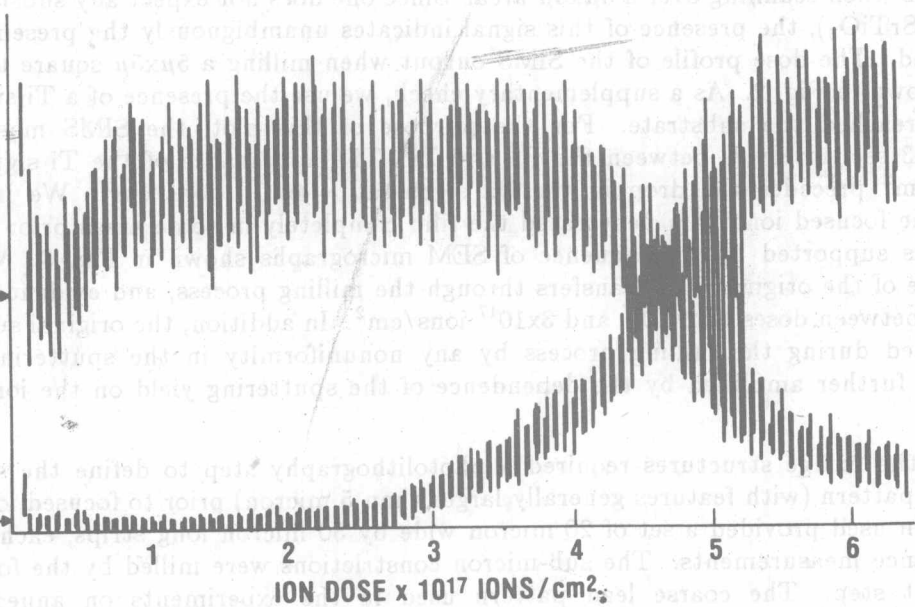


Fig. 2 SIMS end point profile with a 5 $\mu$ x5 $\mu$  square milled into a 2000Å thick film of Ba<sub>2</sub>YCu<sub>3</sub>O<sub>7</sub> film on SrTiO<sub>3</sub> substrate.

DOSE x1017 I/cm2

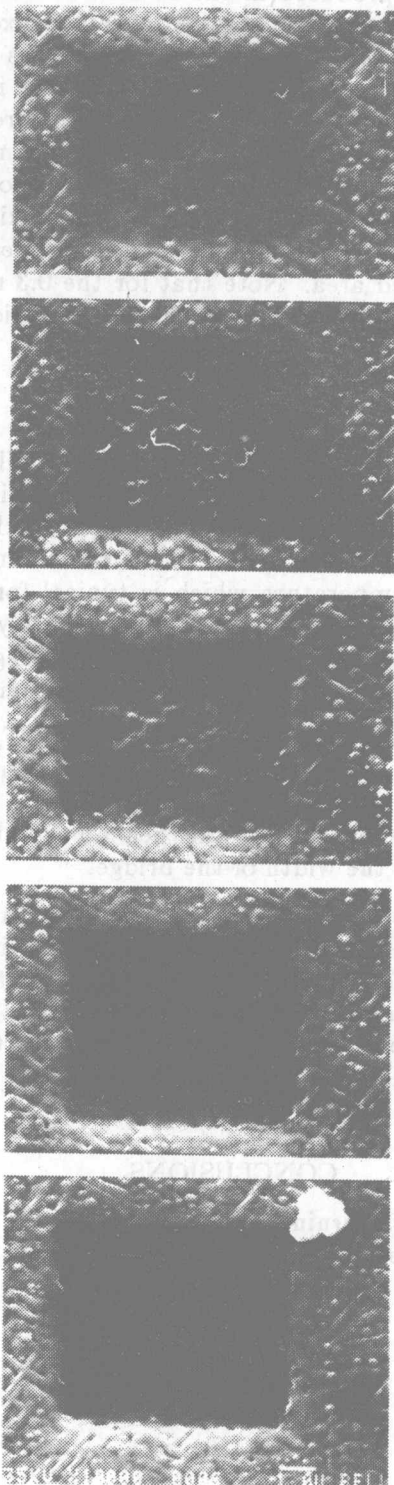


Fig. 3 SEM micrographs depicting the milling process at various ion doses for a 2000Å thick film of Ba<sub>2</sub>YCu<sub>3</sub>O<sub>7</sub> on a SrTiO<sub>3</sub> substrate.

Bridge widths of 0.3 to 5 microns were produced in the 20 micron stripes using the focused ion beam. The process is depicted in Fig. 4. The beam was scanned over a 3-pixel wide band from either edge of the stripe until a  $\sim 0.3$  micron wide cut separating the two banks was produced in the film. The pixel size used was 0.05 micron so that there was considerable leakage into adjacent pixels (a properly normalized profile of the beam is shown in Fig. 4). This ensured that regions of a scanned area received virtually uniform exposure. Given the finite width of the milling beam there is some ambiguity in the specification of the bridge width. For the purpose of this discussion, we define it as the distance between the two points in the calculated dose falls to 50% of its maximum value. The effective width of the bridge may be somewhat smaller depending on the effectiveness of the damage caused by the beam tails to the periphery of the milled area. Note that for the 0.3 micron bridges, the center of the bridge receives a dose of 10% of the milling dose. A series of SEM micrographs of bridges produced in annealed  $\text{Ba}_2\text{YCu}_3\text{O}_7$  is shown in Fig. 5.

## RESULTS AND DISCUSSION

The electrical characteristics of the bridges produced in annealed films are shown in Figs. 6 and 7. Following focused ion beam milling, all bridges demonstrate metallic behavior, with  $R(290\text{ K})/R(90\text{ K})=3$ . The bridges with widths of  $\geq 0.6$  microns shown little or no change in critical temperature; all have  $T_c \geq 88.5\text{ K}$  and transition widths of 1 K. The critical current of these bridges has the approximately linear dependence on temperature which is typical for these films. The slopes of the critical current vs temperature curves in Fig. 7 scale approximately with the width of the bridges. Thus, we conclude that  $J_c$  for the bridges has not been significantly degraded by the milling process and the reduction of the critical current for these bridges is due to their small cross-section.

The 0.3 micron bridges, however, showed a significant decrease in  $T_c$  when compared to the original film, finally reaching  $R=0$  below 40 K. The resistance vs temperature characteristics of these bridges also have rather broad superconducting transitions ( $>50\text{ K}$ ) with long, low resistance tails. We conclude that the peripheral exposure caused by the beam current distribution (tails) produces a damaged region in the film which spans the width of the bridge.

A series of experiments were also performed on as-deposited films (that is, prior to oxygen annealing). These films are amorphous and quite smooth (and presumably uniform). It was thought that the results of milling fine features could be made more reproducible if it was done on a uniform smooth substrate. We found that once an unannealed film was exposed to the Ga ion beam, it would anneal into a poor quality film with non-uniform characteristics. This was true even when the ion dose was limited only to that for imaging (much less than  $10^{14}/\text{cm}^2$ ). We speculate that low levels of Ga implanted before the anneal are sufficient to poison these films.

## CONCLUSIONS

We have demonstrated a submicron patterning method for high  $T_c$  superconductor thin films which does not degrade the original properties of the material.

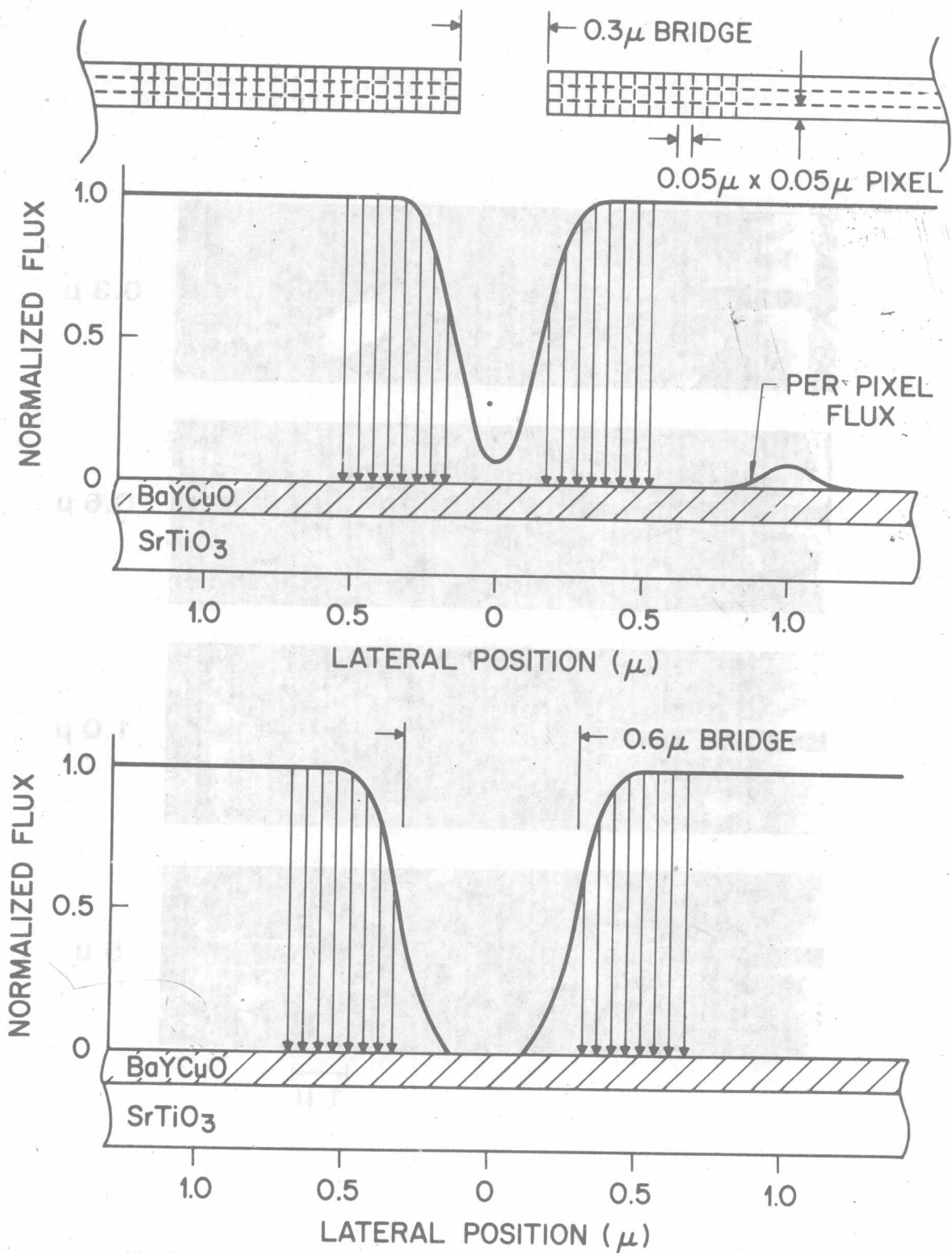


Fig. 4 Calculated ion dose profiles for milling various sized bridge structures for a 0.2  $\mu$  FWHM ion beam.



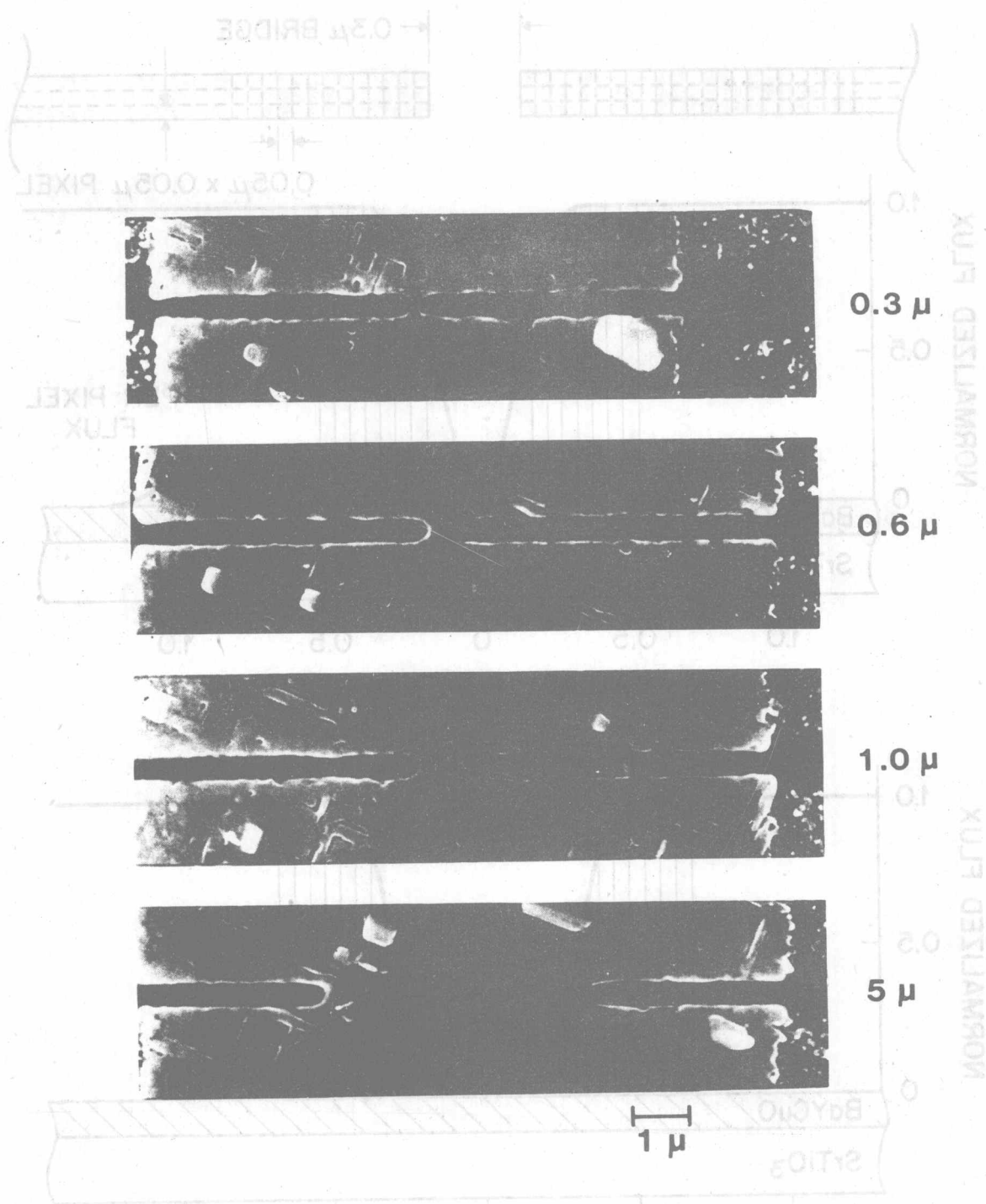


Fig. 5 SEM micrographs of bridges milled in annealed  $\text{Ba}_2\text{YCu}_3\text{O}_7$  films.