

# Gallium Ion Implantation into Niobium Thin Films Using a Focused-Ion Beam

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**Abstract**— We have implanted 30 kV gallium ions into niobium films 100 Å thick using a focused-ion beam (FIB). The nature of the FIB tool allows the irradiation of only a specific, controllable, area of the substrate, from hundreds of square microns down to an arbitrary, user-defined pattern of 5E-5 square micron or less. A sacrificial layer of gold covering the niobium controls the range of the incident gallium ions and prevents the niobium film from sputtering away under bombardment. This article examines this behavior phenomenologically, including information about the changes in transition temperature and resistance of the implanted samples. Also a curious unexplained feature of the resistive transition at implant doses below about  $3 \times 10^{19} \text{ cm}^{-3}$  will be presented and discussed.

**Index Terms**—Niobium, Thin Film, Ga, Ion Implantation, Focused-Ion Beam.

## I. OVERVIEW

Ion implantation into niobium films has been a subject of interest for at least thirty years [1-4]. The dopants which have been investigated include argon, iron, nitrogen, and gadolinium, using implant energies between 20-150 keV. Mechanisms for  $T_c$  suppression by implantation discussed in the referenced literature include lattice modification, the proximity effect, and the effects of magnetic impurities (Gd).

This work involves implantation of Ga into thin (100 Å) niobium films. The properties of superconducting binary Nb-Ga alloys have been known for many years. [5,6] It has also been shown that Ga doping of Nb<sub>3</sub>Sn conductors can have beneficial effects. [7]

Attempts to fabricate niobium diffusion-cooled hot-electron bolometers (HEBs) using a Ga<sup>+</sup> focused-ion beam (FIB) [8] have motivated the present research. Since in this instance the implantation of gallium into niobium is essentially a problem of contamination, the focus has been largely to understand and quantify the effects of the contamination. Two factors, however, deserve special mention.

Firstly, the FIB is an extraordinarily flexible, controllable, and fine (with a spot size as small as 7 nm) tool which could

be used to implant geometries not possible to create by any other method. Secondly, a surprising feature of the resistive transitions of implanted samples lacks a simple explanation, and may be of interest to other researchers.

## II. DESCRIPTION OF THE EXPERIMENTAL TECHNIQUE

Films of 10nm Nb covered by a layer of gold (10 or 30 nm) were deposited under zero-stress conditions under a single vacuum onto quartz wafers using DC magnetron sputtering and patterned using liftoff. An in-situ ion clean is used before deposition. The base pressure of the sputtering chamber was in the mid-10<sup>-8</sup> Torr range. The samples exhibited a resistance ratio R(300 K)/R(10 K) slightly greater than two.

The actual sample structures for implantation and test consisted of two 3 mm x 1.5 mm contact pads, connected in the middle by a bridge 10 μm wide by 5 μm long. A negative photoresist, intended for implantation applications, covered all of the areas of the samples except for an open window over part of the small bridge and the open substrate field on either side of it, so that only about one square of film about 15 μm across was irradiated during the implantation experiments. After FIB implantation, the resist was removed with acetone; the gold layer was not removed at any time.

The FIB was used to implant gallium into the samples simply by focusing on the open window in the photoresist for a set length of time. The process is not very exact for short exposures, due to the need to find and focus on the area to be exposed with the same Ga beam to form the screen image. However, this problem will not be difficult to overcome in future work, especially if user-defined patterns of implantation are investigated. Aside from the exposure time, the thickness of the sacrificial gold layer and the FIB beam current and magnification all served to control the dose implanted in the exposed section of film. The energy of the implanted gallium ions was 30 kV.

## III. THEORETICAL ANALYSIS USING SRIM

SRIM [9] was used to develop predictions regarding the distribution of gallium dopants within the quartz/Nb/Au structure. The predicted range of 30 kV Ga ions in gold is 95 Å, with a 56 Å straggle. The range increases to 109 Å quartz/Nb(10nm)/Au(10nm) structure because of the greater range of the energetic gallium ions in niobium and quartz, as compared to gold. Simply by counting the number of ions in

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the proper range of depths, one concludes that 35% of the ion flux incident upon this structure will be caught in the niobium layer. In the experiments which are described below, this implies a Ga impurity concentration in the Nb film on the order of  $10^{19} \text{ cm}^{-3}$ .

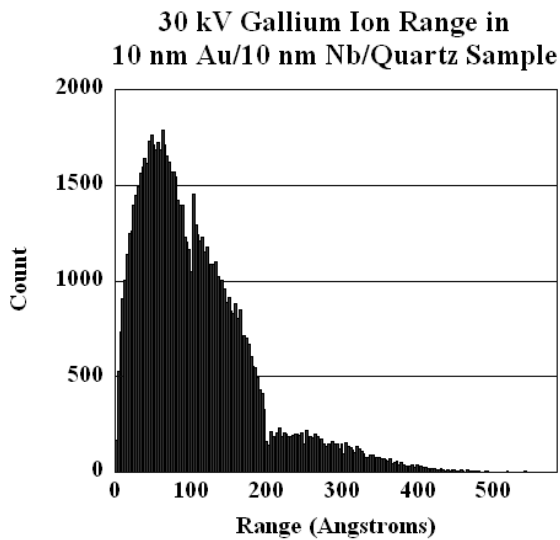


Fig. 1. Distribution of implanted gallium, according to an SRIM simulation utilizing  $10^5$  ions in 2 Å bins. The niobium layer is between 100 and 200 Å deep in this histogram. Analysis of this data reveals that about 35% of the ion flux incident on the sample will stop in the Nb layer, although clearly its distribution within that layer is not uniform.

Figure 2 shows the calculated effects of changing the thickness of the sacrificial layer of gold. The percentage of the ion flux captured within the niobium film decreases by about two orders of magnitude, to nearly zero, as the gold thickness increases from 100 to 300 Å.

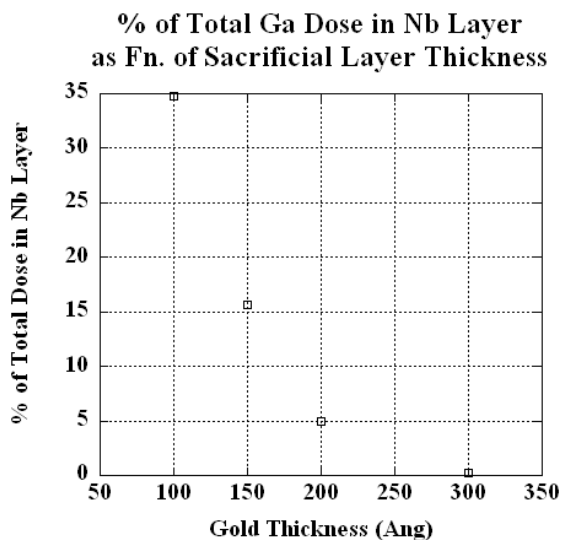


Fig. 2. Capture percentages of incident Ga flux within the 100 Å Nb layer depend strongly upon the thickness of the gold layer on top, according to simulations with SRIM using  $10^5$  ions.

#### IV. EXPERIMENTAL RESULTS

Figure 3 shows the results of the experiments outlined earlier on a Nb(10nm)/Au(10nm) film on quartz. The top two curves were implanted with a 13 pA beam current, with a magnification of 10,000x (corresponding to a field of view of 29 microns square), for periods of 60 and 30 seconds, respectively. The third curve (with a dose of  $27 \times 10^{18} \text{ cm}^{-3}$ ) was implanted at the same magnification, but with a lower 4 pA beam current for a time of 25 seconds. This curve does not show the entire transition of the sample- the 10K resistance of the sample is  $20 \Omega$  due to a third soft superconducting transition above 7K. The nature of the remainder of this transition above 7K is not understood. Finally, a sample which did not undergo implantation is included for reference.

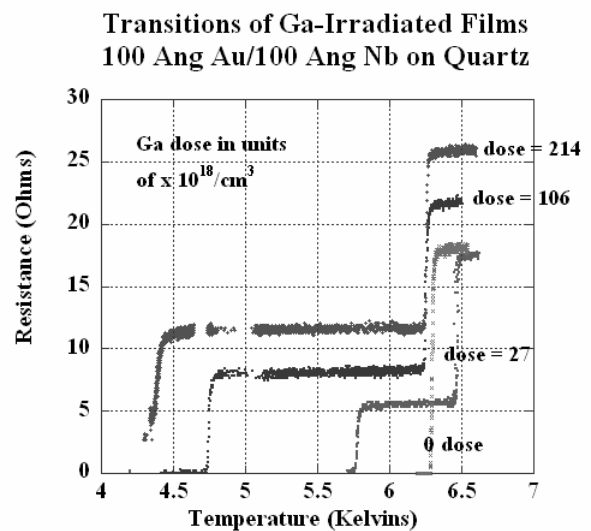


Fig. 3. Results of the implantation experiments. The curve for the sample irradiated with a dose of  $27 \times 10^{18} \text{ cm}^{-3}$  exhibits an odd feature: the portions of the sample which were not irradiated actually show an increase in  $T_c$  as a result of the implantation.

For the most part, the data presented are easy to interpret. The portions of the samples in which gallium is implanted clearly seem to show a decrease in transition temperature and an increase in resistance of the irradiated area, while other portions of the sample (the test pads) do not change, creating a transition in two steps. The curve corresponding to a dose of  $27 \times 10^{18} \text{ cm}^{-3}$ , however, shows the transition temperature, of what we assume is the non-intended irradiated portion of the sample, actually increasing. A similar effect was first noticed previously on a different set of samples which did not have the protective resist. That set of samples was irradiated, with an 11 pA beam current and a magnification of 1200x, resulting in doses between 3 and  $21 \times 10^{18} \text{ cm}^{-3}$ . The resulting curves showed the same trends of decreasing  $T_c$  and increasing resistance, but every curve also showed a  $\sim 0.15\text{K}$  increase in the transition for the 2<sup>nd</sup> step, representing what we assumed was an effect on the non-intended irradiated portion of the sample from a thinning of the overlying Au from incidental flux. For the present case, however, the test pads

are covered with resist, so it is unclear how the Ga flux could still be affecting the test pads and only for low applied ion fluxes.

Some preliminary experiments on Nb(10nm)/Au(30nm) samples have also been undertaken, but without definitive results. In some cases, an implantation dose on the order of  $10^{16} \text{ cm}^{-3}$  seemed to simply shift the transition curve upwards by about 0.05 K. A likely explanation is that the FIB removed some of the open gold over the Nb during implantation, reducing the proximity effect and thereby increasing its critical temperature. The thicker 30nm Au film, however, appears to have prevented any effects to the underlying Nb layer

A summary of the gallium exposure results is given in Figure 4.

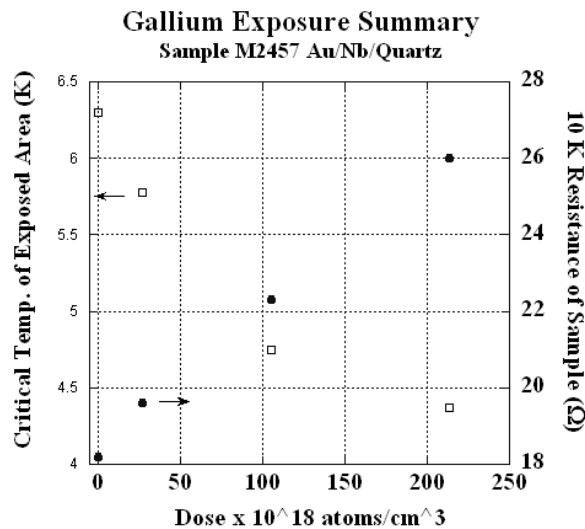


Fig. 4. Summary of the gallium implantation experimental results, on Nb(10nm)/Au(10nm) films. The open squares detail the suppression of critical temperature with increasing dose; the closed circles, the increase in sample resistance. Since the gold was not removed from the sample, it is not certain that the second effect is not at least in part due to removal of the gold by the FIB during implantation.

## V. CONCLUSION

We have examined the decrease in transition temperature and (in part) increase in resistance which a 10 nm Nb film undergoes as a result of ion implantation with gallium using a focused-ion beam. It is possible that a portion of the increase in resistance with dose documented here is a result of the removal of a portion of the sacrificial layer of gold by the FIB, although there is not visual evidence of substantial Au removal for this to be a significant effect. Future experiments in which the gold is removed with a wet etchant can resolve this question.

The possibility of using the user-programmable FIB to implant specific patterns over the surface of a niobium film, creating for instance a meandering pattern or a series of weak links, has been raised, though not examined. An interesting possibility, from the perspective of the work with HEBs which motivated this investigation, involves the possibility of the

FIB trimming of devices, which would be especially interesting for applications where an array of devices has been fabricated and their uniformity is a principal concern. For this application, implantation into a Nb thin film under a sacrificial layer of passivating Ge would be of interest, and that possibility has not been investigated. Another possible application is using Ga implantation to specifically lower the transition temperature, which had been shown to increase mixer performance [10].

Finally, the curious behavior of the non-irradiated portion of the sample at doses below  $3 \times 10^{19} \text{ cm}^{-3}$  has been presented. No explanation for this increase in the transition temperature of what appears to correspond to the test pads is offered at this time.

## REFERENCES

- [1] P. Crozat and R. Adde, "Critical Temperature and Density of States in Ion Implanted Nb Films", *Physica* 108B, pp. 1251-1252, 1981.
- [2] C.H. Arrington III and B.S. Deaver Jr., "Superconducting Weak Links formed by ion implantation", *Applied Physics Letters*, Vol. 26, No. 4, pp. 204-206, 1975.
- [3] C. Camerlingo, P. Scardi, C. Tosello, and R. Vaglio, "Disorder effects in ion-implanted niobium thin films", *Physical Review B*, Vol. 31, No. 5, pp. 3121-3123, 1985.
- [4] P.D. Scholten and W.G. Moulton, "Effect of ion-implanted Gd on the superconducting properties of thin Nb films", *Physical Review B*, Vol. 15, No. 3, pp. 1318-1323, 1977.
- [5] J. Sosnowski, M. Drys, and J. Szymaszek, "Properties of Superconducting Nb-Ga Alloys with A15 Type Structure", *Phys. Stat. Sol. (a)* 42, pp. K159-K164, 1977.
- [6] S. Foner, E.J. McNiff, Jr., G.W. Webb, L.J. Vieland, R.E. Miller, and A. Wicklund, "Upper Critical Fields of  $\text{Nb}_x\text{Ga}_{1-x}$ : A Binary High Temperature Superconductor", *Physics Letters*, Vol. 38A, No.5, pp. 323-324, 1972.
- [7] M. Rudziak and T. Wong, "Development of a  $\text{Nb}_3\text{Sn}$  Conductor Containing Ga and Mg Dopants", *IEEE Transactions on Applied Superconductivity*, Vol. 11, No. 1, March 2001.
- [8] A. Datesman, J. Schultz, A. Lichtenberger, D. Golish, C. Walker, and J. Kooi, "Fabrication and Characterization of Niobium Diffusion-Cooled Hot-Electron Bolometers on Silicon Nitride Membranes", this proceedings.
- [9] "The Stopping and Range of Ions in Matter", [www.srim.org](http://www.srim.org).
- [10] I. Siddiqi, D. E. Prober, "Nb-Au bilayer hot-electron bolometers for low-noise THz heterodyne detection," *Applied Physics Letters*, Vol. 84 pp.1404, 2004.