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

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ABSTRACT

We have implanted 30 keV 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 x 10^{19} cm^{-3} will be presented and discussed

Gallium Ion Implantation

Ion implantation into niobium films has been a subject of interest for at least thirty years. The dopants which have been investigated include argon, iron, nitrogen, and gadolinium, using implant energies between 20-150 keV. This work involves implantation of Ga into thin (100 Å) niobium films.

Attempts to fabricate niobium diffusion-cooled hot-electron bolometers (HEBs) using a Ga+ focused-ion beam (FIB) 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. Films of 100 Å Nb covered by a layer of gold (100 or 300 Å) were deposited under zero-stress conditions under a single vacuum using DC magnetron sputtering and patterned using liftoff. The samples were deposited on a quartz substrate and cleaned prior to deposition with an in-situ ion mill.

SRIM [9] was used to develop predictions regarding the distribution of gallium dopants within the quartz/Nb/Au structure. The predicted range of 30 keV Ga ions in gold is 95 Å, with a 56 Å straggle. The range increases to 109 Å in a 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 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} cm^{-3}.

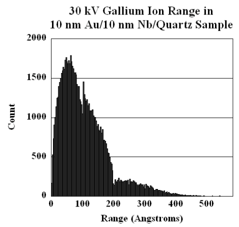

Fig. 1. Distribution of implanted gallium, according to an SRIM simulation utilizing 105 ions in 2 Å bins. The niobium layer is between 100 and 200 Å deep in this histogram. Analysis of this data reveals that about 35% of the ion flux incident on the sample will stop in the Nb layer, although clearly its distribution within that layer is not uniform.

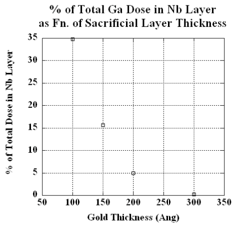

Fig. 2. Capture percentages of incident Ga flux within the 100 Å Nb layer depend strongly upon the thickness of the gold layer on top, according to simulations with SRIM using 105 ions.

Device Testing

The actual sample structures for implantation and test consisted of two 3 mm x 1.5 mm pads, connected in the middle by a bridge 10 mm wide by 5 mm 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. The samples exhibited a resistance ratio R(300 K)/R(10 K) somewhat greater than two. 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 energy of the implanted gallium ions was 30 kV.

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 x 10^{18} cm^{-3}) was implanted at the same magnification, but with a a lower 4 pA beam current for a time of 25 seconds. This curve does not show the entire transition of the sample- the 10 K resistance of the sample is 20 Ω 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.

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 x 10^{18} 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 x 10^{18} cm^{-3}. The resulting curves showed the same trends of decreasing Tc and increasing resistance, but every curve also showed a ~0.15K increase in the transition for the 2nd 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 effecting the test pads and only for low applied ion fluxes.

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

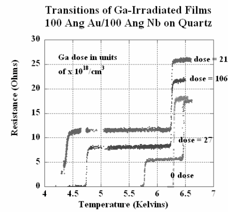

Fig. 3. Results of the implantation experiments. The curve for the sample irradiated with a dose of 27 x 1018 cm-3 exhibits an odd feature: the portions of the sample which were not irradiated actually show an increase in Tc as a result of the implantation.

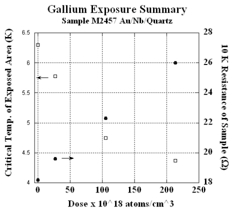

Fig. 4. Summary of the gallium implantation experimental results, on 10 nm Au/10 nm Nb films. The open squares detail the suppression of critical temperature with increasing dose; the closed circles, the increase in sample resistance. Since the gold was not removed from the sample, it is not certain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

SUMMARY

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 that question. The curious behavior of the non-irradiated portion of the sample at doses below 3 x 10E^{19} cm^{-3} has been presented, although no explanation is currently offered. It is possible to use the user-programmable FIB to implant specific patterns over the surface of a niobium film and to trim HEB devices, which would be especially interesting for applications where an array of devices has been fabricated and their uniformity is a principal concern. Also possible is the use of the FIB to specifically decrease Tc, which has been shown to improve mixer performance.

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