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J.Y. ROBIC
J. PIAGUET
D. DURET
J.C. VELER
** J.L. VERAN
D. ZENATTI

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D'ELECTRONIQUE
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CENTRE D'ÉTUDES NUCLÉAIRES
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** Now at I.B.M. - 06610 LA GAUDE
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Résumé

ION IMPLANTATION IN SUPERCONDUCTING [*] NIOBIUM AND Nb_3 Sn THIN FILMS : ADJUSTMENT OF JOSEPHSON MICROBRIDGES AND SQUID DEVICES

J.Y. ROBIC J. PIAGUET D. DURET J.C. VELER J.L. VERAN[**] D. ZENATTI

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In this paper the principles of operation of Josephson junctions and Squids are resumed. An ion implantation technique for the adjustment of the critical current is presented.

High quality superconducting thin films were obtained by electron gun evaporation of niobium on heated substrates. Polycrystalline Nb_3 Sn was made by annealing (1000 K, 10^{-6} Torr) a multilayer structure of successively evaporated niobium and thin films.

Selected ions (helium, neon, argon) were implanted at doses ranging from 10^{13} to 10^{17} cm^{-2}. After implantation the critical temperature, the critical current and the normal resistivity were measured on special photoetched geometries. The variations of these electrical properties depend on the nuclear energy loss. The critical temperature of Nb_3 Sn is decreased by ion implantation and can be increased again by a new annealing.

The parameters of the ion implantation were defined in order to obtain a critical temperature slightly higher than the operating temperature. The geometries of the microbridges and the implanted areaswhere then chosen to obtain appropriate criticals currents ($\sim$ 10 µA) at the operating temperature. The obtained microbridges were used as junction elements in superconducting quantum interference devices (SQUID)

INTRODUCTION

Ion implantation is a possible method for modifying physical characteristics of superconducting metals and alloys (1).

We describe in this paper the realization of thin film Nb and Nb_3Sn Josephson junctions using ion implantation as an adjustment technique.

Josephson junctions, obtained by a weak coupling between two superconductors, may be realized in several ways :

- with bulk superconductors : point contacts
- using thin film technology : tunnel jonction, proximity effect junctions, microbridges.

In the case of microbridges (Fig. 1), the required dimensions to obtain Josephson coupling are very small (less than 1 µm). For this reason, microbridges are preferably etched with reproductible dimensions of 5 µm x 5 µm ; the superconductivity is then locally weakened either by local annealing using current pulses (2) or diminution of the dimensions by anodization for example (3) or using ion implantation.

The field of applications of devices based on Josephson effect is very large : microwave detection, voltage standard, ultra fast logic, magnetometry. It is thus important to be able to produce large quantities of Josephson junctions in a reproducible way. For this reason, thin film photoetched devices are of great interest.

The principal parameters that must be adjusted in the fabrication process are :

- <u>Operating temperature and critical current</u>. It is necessary to decrease the superconducting transition (critical temperature) of the microbridge to a value slightly above the operating temperature in order to obtain a reasonably small critical current (10 µA to 100 µA). Generally, the liquid helium boiling temperature (4.2 K) is chosen as the operating temperature. It is an important constraint, since the variation of the microbridge critical current as a function of the temperature is very large in this temperature range (Fig. 6). In addition, small variations of the dimensions can cause large variation of the critical current.

In some applications, the microbridges are used at higher operating temperatures (8-18°K) maintened by the so-called cryogenerators. In this case, the operating temperature can be adjusted and the variation of the critical temperature due to the adjustment procedure, for a given microbridge, is less critical.

- Normal state resistance. The normal state resistance of the microbridge is measured slightly above the superconducting transition. A good microbridge (which produces a low noise) has a high normal state resistance ($\gtrsim$ 10 Ω).

  These resistance values can be obtained by decreasing the physical dimensions. The choice of the adjustment method is also an important parameter.

The different steps of fabrication of a microbridge are thus :

. deposition of a good quality superconducting thin film (highest possible critical temperature).

. Photoetching of a microbridge structure in this film.

. Weakening of the microbridge superconductivity, i.e. decrease of the critical temperature to a value slightly above the operating temperature. The critical current is simultaneously decreased to a correct value at the operating temperature.

The last step can be done using ion implantation. This method, which induces defects in the cristalline structure, weakens the superconductivity especially in the case of strongly coupled superconductors.

The niobium ($T_c \simeq 9.2$. K) and the compound Nb_3Sn (T_c max = 18.5°K, A15 structure) fall in this category.

The ion implantation used as an adjustment technology has the following advantages :

- an easier fabrication process of the microbridges faciliting a collective treatment

- a precise control of the microbridge characteristics (normal state resistance and critical current)
- a process independent of the thermal conductivity of the substrate.

EXPERIMENTAL CONDITIONS

High quality superconducting niobium thin films were obtained using an electron gun evaporation on a heated substrate (400°C) in high vacuum (10^{-8} Torr). After niobium evaporation, the substrate was cooled down and a gold film was deposited on the niobium film in the same vacuum enclosure.

The purpose of this gold layer was to make electrode contacts and to protect a part of the niobium against the ion implantation (Fig. 1).

We obtained 1200 Å niobium films with a high critical temperature (9.2°K) and a good resistivity ratio :

$$R = \frac{\rho_{300K} - \rho_{10K}}{\rho_{10K}} = 3{,}6$$

Three sample shapes were then photoetched on the substrate using standard techniques :

- On plane substrates : (SiO_2 or $A\ell_2O_3$)
  1. A resistive shape in order to measure the critical temperature (T_c) and the resistivity ratio.
  2. Microbridges with different geometries (Fig. 1) (ℓ and L)
- On cylinders (SiO_2)
  3. Microbridges in series with a superconducting inductance (Fig. 2). These special devices (called SQUIDS (4)) quantize the magnetic flux (the flux quantum is $\emptyset_o = 2{,}07\ 10^{-15}$ Wb) in the superconducting inductance. They are able to measure magnetic fields with a sensibility which reaches 10^{-14} T. $Hz^{-1/2}$

Three ions were implanted : Ne^+, Ar^+, He^+, at a single energy or at two energies to obtain more uniform damage from the top to the bottom of the films. The doses indicated in the figures are the sums of the implanted doses. The vacuum during the implantation was under 10^{-6} Torr and the ion current was limited in order to limit the irradiance to the value of 10^3 $W.m^{-2}$.

RESULTS AND DISCUSSIONS

Niobium

Before and after implantation of neon, the critical temperature, the room remperature resistivity (ρ_{300K}) and the resistivity just before the superconducting transition (ρ_{10K}) of niobium thin films were measured. These values versus ion doses are given in Figures 2 and 3.

A very high dose of neon, 2 to 3 10^{17} cm^{-2}, was necessary to decrease the initial critical temperature of niobium to a temperature near the liquid helium temperature (4.2K). The resistance was multiplied by a factor of 6.

Figure 4 shows the variation of the critical temperature as a function of the average density of energy loss by nuclear collisions (P) of He, Ne and Ar. This energy loss is defined as :

$$P = \frac{\sum En_i D_i}{e}$$

where e = thickness of the film

En_i = energy loss by nuclear collisions

D_i = dose number i

We feel that the decrease induced by implantation of inert gases is only due to defects created by nuclear collisions and is independent of the nature of the implanted ions.

The ion implantation parameters and the nature of the implanted ions have no influence on the relation between critical temperature and resistivity ratio according to the law found by De Sorbo (5) (Fig. 5) :

$$T_c = 9.46 - 2.48 \quad \frac{\rho_{10K}}{\rho_{300K} - \rho_{10K}} \qquad (\text{for } T_c > 6K)$$

We can notice, in Fig. 6, the steep slope of the curve $I_c(T)$ for low values of the critical current. This implies that adjustment of a microbridge by ion implantation on niobium for 4.2K operation is particularly critical. A critical current of 40 μA for a 5 μm wide microbridge requires an adjustment of T_c to 5% above the operating temperature.

In Table 1, the critical current for variable width and fixed critical temperature (5.2K) microbridges are listed. The operating temperature was 4.2K. As one could think the critical current is a function of the width (ℓ) of the microbridges. Nevertheless the reproductibility of the results is not very good. Remark that we obtain Josephson coupled microbridges having a length (L) as great as 11 µm.

The normal resistance of the fabricated microbridges was between 0,85 Ω and 9,5 Ω depending of the geometry of the microbridges.

	l µm	L µm	Ic mA	Rn Ω
1	2,2	11	0,02	
2	6,5	11	0,8	9,5
3	6,5	11	2	9,5
4	15	3	4	2
5	15	3	8	1,4
6	23	3	7	1,2
7	21,5	3	8	0,85

TABLE 1

Nb_3Sn

We present in Figure 5 the correlation between the critical temperature and the resistance ratio of Nb_3Sn films before and after ion implantation. It is well known that the resistance ratio is strongly affected by defects, so from the results of Figure 5 we may conclude that defects induced by ion implantation are of utmost importance in controlling the T_c in Nb_3Sn.

Fig. 7 shows the measured ΔT_c as a function of the total number of implanted ions per unit area. The effects of damage on T_c of Nb_3Sn were found to be stronger than those observed in Nb. For example, one degree decrease of T_c needs a 10^4 higher dose of neon in Nb than in Nb_3Sn.

The ion implanted microbridges behaved like the ones (6) realized using pulse adjustment technique (I(V) curve, microwave characteristics).

Critical current vs temperature for two microbridges implanted with the same dose are plotted in Fig. 8. One can remark that the slope near the transition temperature is less steeper for the smaller microbridges. This confirms the interest of the small microbridges (less than 5 µm).

We annealed adjusted microbridges (T_c less than 4.2K) in the conditions of Nb_3Sn fabrication (30 minutes, 1000°C, 10^{-7} Torr). The critical temperature of the microbridge increased to a value of 12K after a first annealing and to 14K after a second one. All these microbridges showed a Josephson characteristic.

This adjustment method for the critical current and the operating temperature was applied in the fabrication of SQUIDS. We were able to obtain several SQUIDS working at different temperatures between 8K and 13.5K. The stability was excellent and tested over a year.

CONCLUSION

Further characterization studies are in process in order to evaluate the main parameters concerning the devices sensitivity. These parameters will be used to define a reproducible method of mass production.

This study showed the great interest of the ion implantation used as an adjustment technique for Nb and Nb_3Sn thin film devices. The method was found to be very promising and versatile especially in the case of Nb_3Sn.

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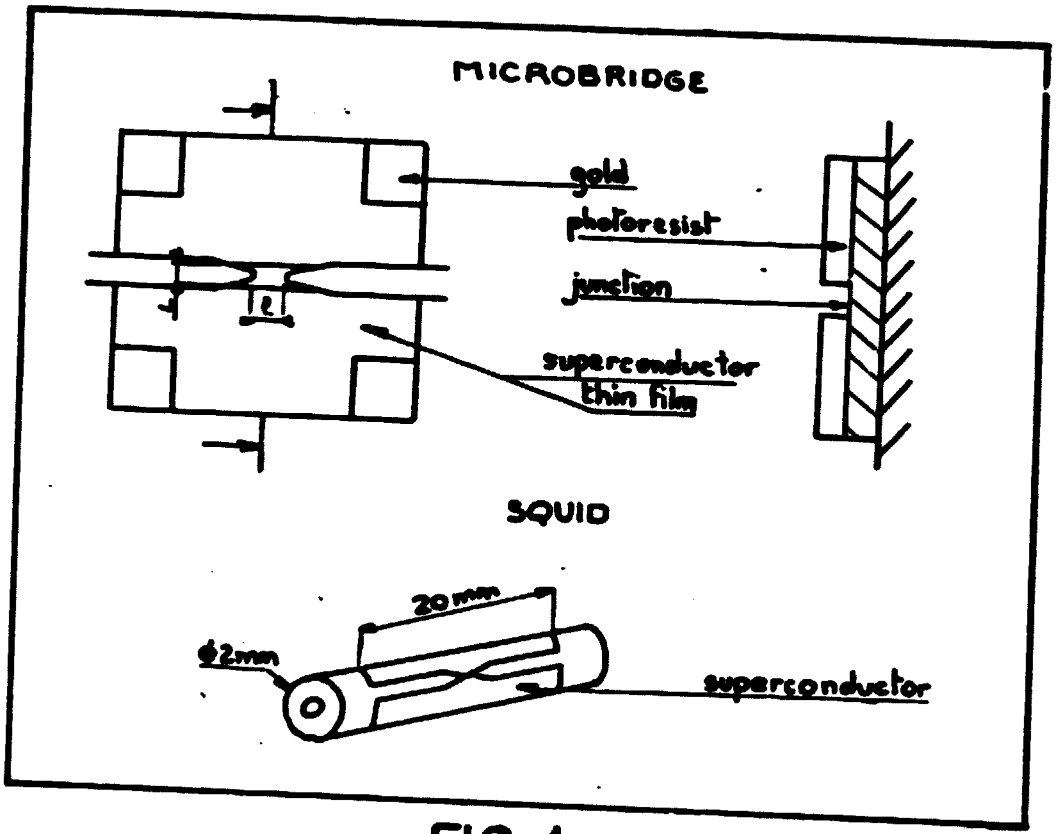
MICROBRIDGE
gold
photoresist
junction
l
superconductor thin film
SQUID
20mm
ϕ2mm
superconductor

FIG. 1

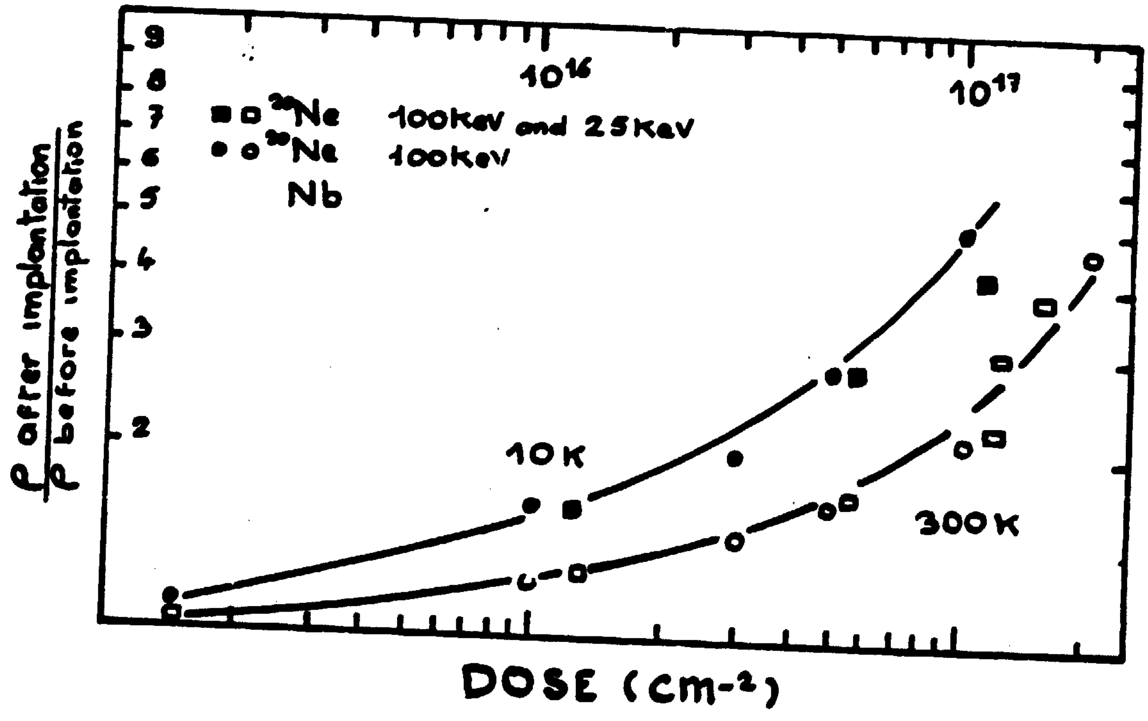
9
8
7
6
5
4
3
2
10^{16}
10^{17}
■ □ ^{20}Ne 100keV and 25KeV
● ○ ^{20}Ne 100keV
Nb
10K
300K
ρ after implantation / ρ before implantation
DOSE (cm^{-2})

FIG. 2

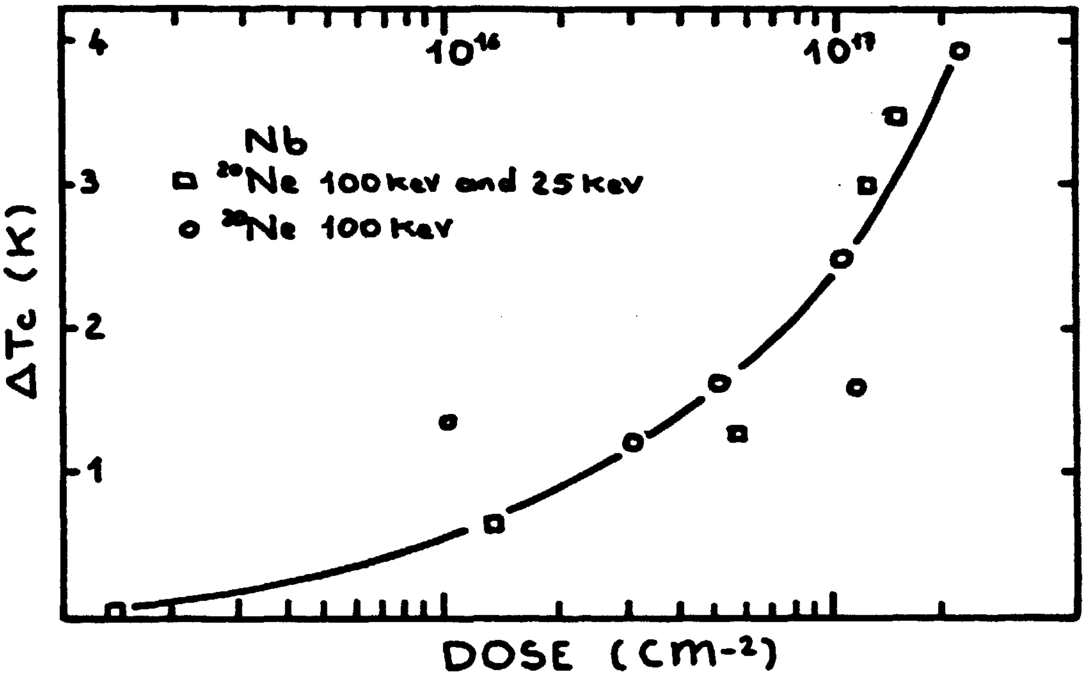
Nb
□ ^{20}Ne 100 keV and 25 keV
○ ^{20}Ne 100 keV
4
3
2
1
10^{16}
10^{17}
ΔTc (K)
DOSE (cm^{-2})

FIG. 3

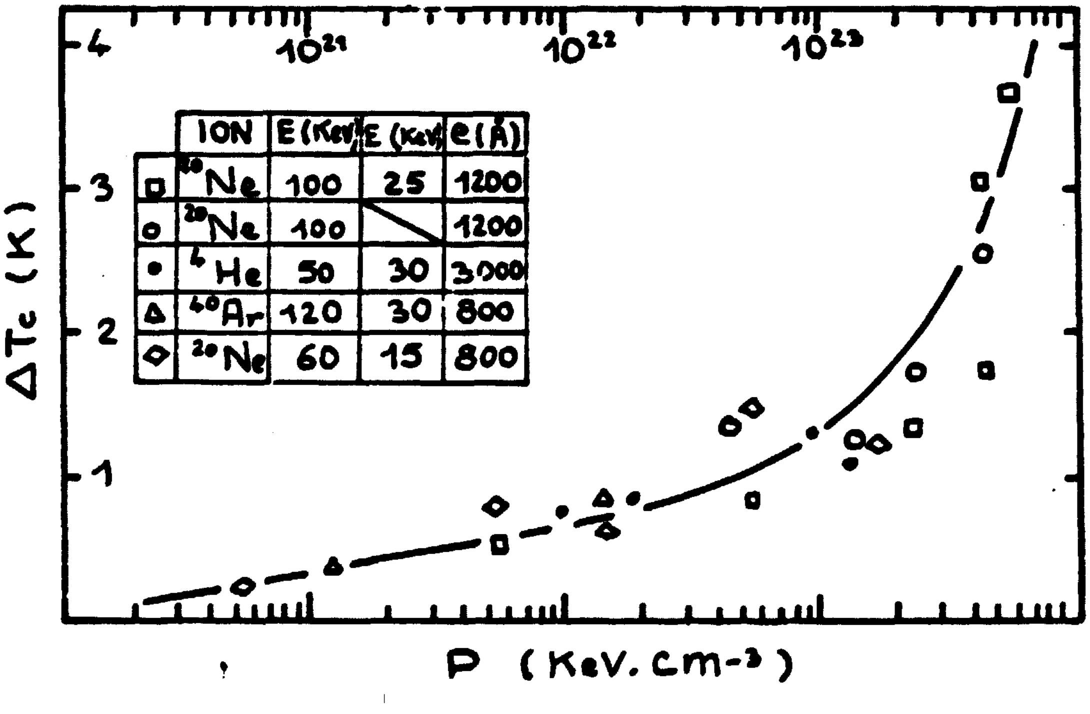
4
3
2
1
10^{21}
10^{22}
10^{23}
ION | E (KeV) | E (KeV) | e (Å)
□ | ^{20}Ne | 100 | 25 | 1200
○ | ^{20}Ne | 100 | | 1200
• | ^{4}He | 50 | 30 | 3000
Δ | ^{40}Ar | 120 | 30 | 800
◇ | ^{20}Ne | 60 | 15 | 800
ΔTc (K)
P (KeV.cm^{-3})

FIG. 4

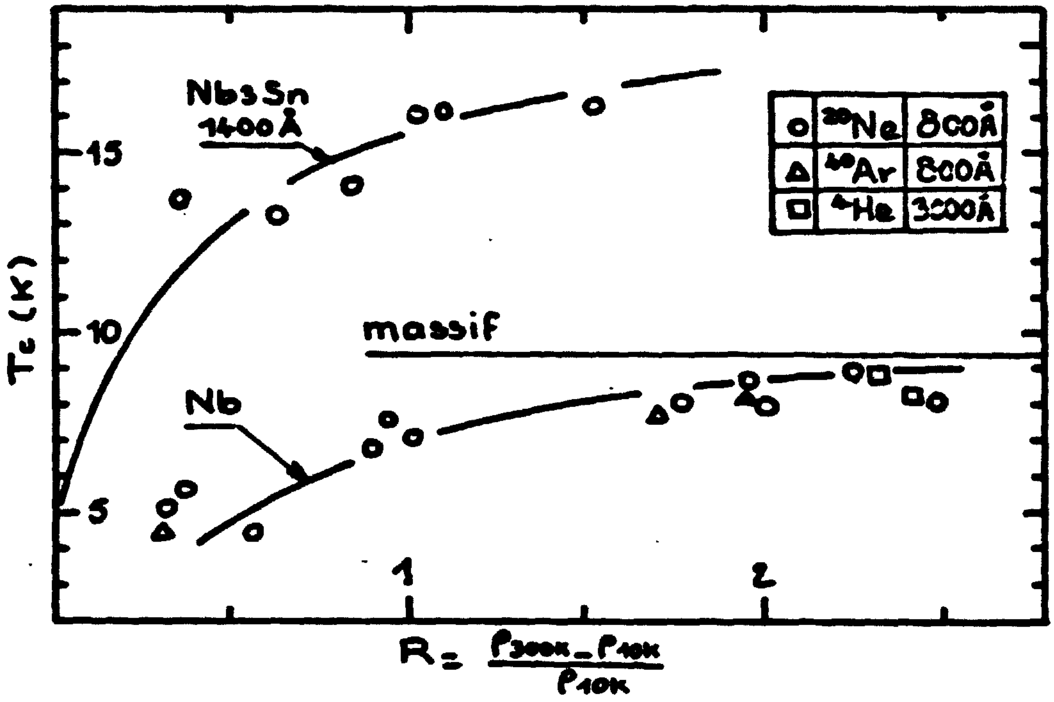
Nb3Sn
1400 Å
○ 20Ne 800 Å
△ 40Ar 800 Å
□ 4He 3000 Å
massif
Nb
Tc (K)
5
10
15
1
2
R = (ρ300K − ρ10K) / ρ10K

FIG. 5

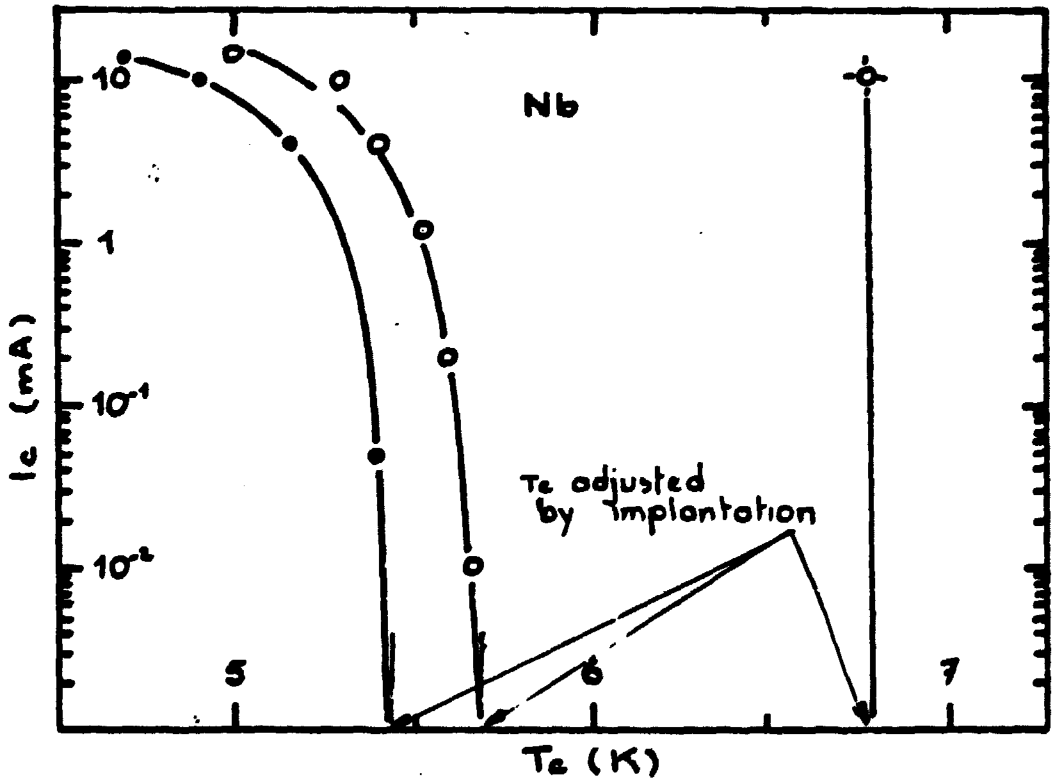
Nb
Tc adjusted by implantation
Ic (mA)
10
1
10⁻¹
10⁻²
5
6
7
Tc (K)

FIG. 6

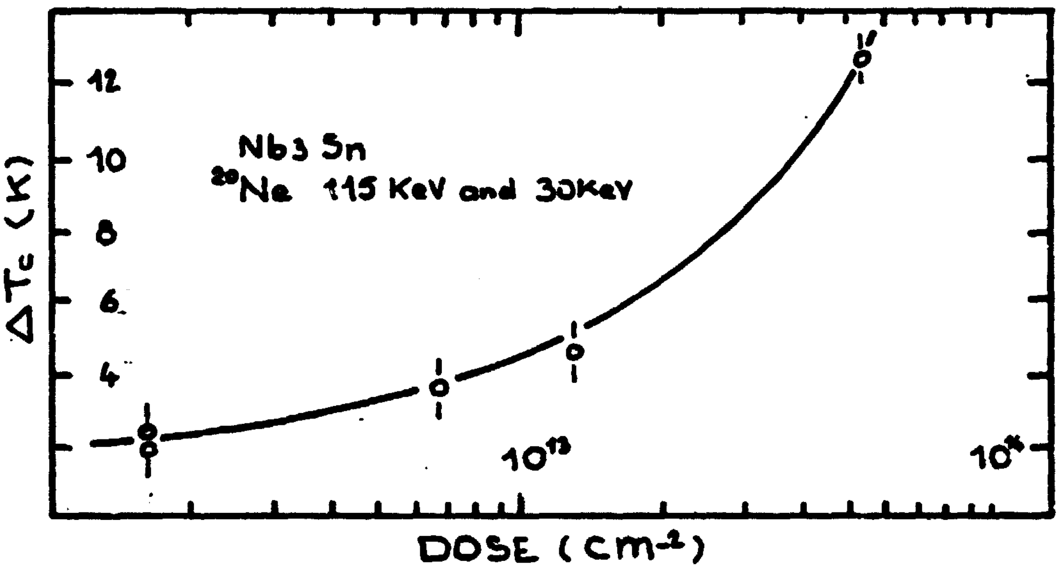
ΔTc (K)
12
10
8
6
4
Nb3 Sn
20Ne 115 KeV and 30KeV
10^13
10^14
DOSE (cm-2)

FIG.7

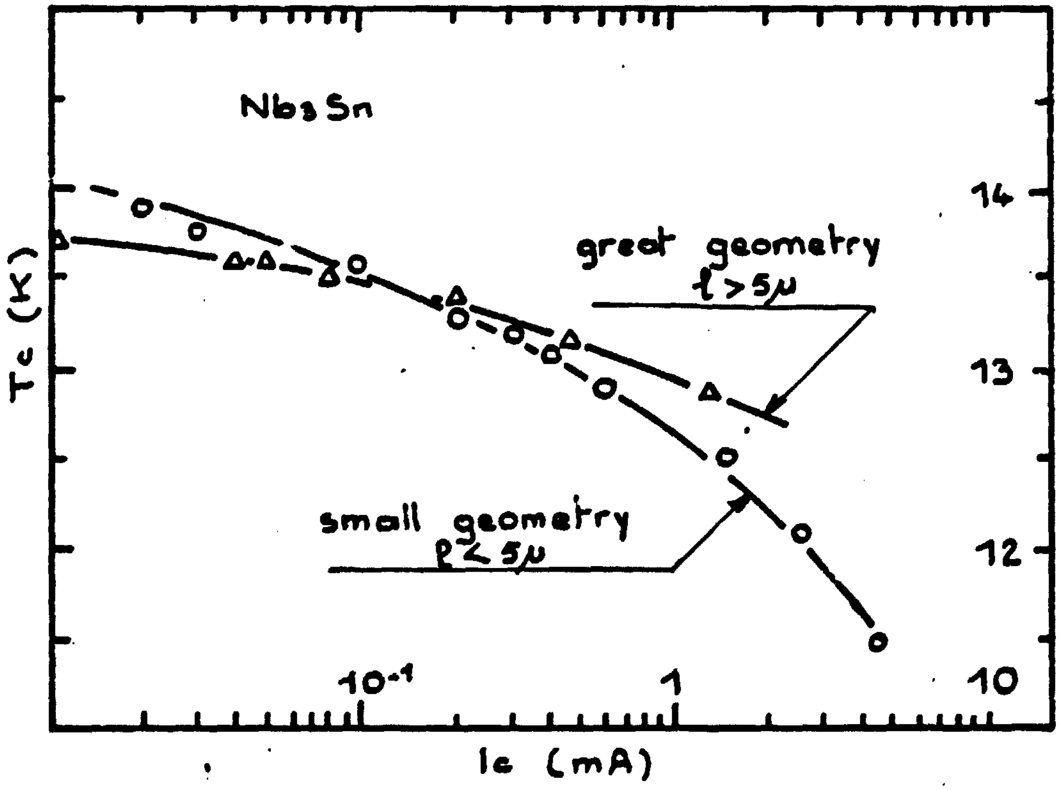
Nb3Sn
Tc (K)
14
13
12
10
great geometry
l > 5μ
small geometry
l < 5μ
10^-1
1
Ic (mA)

FIG.8