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Abstract One of the goals of LUMINEU is to develop NTD-Ge sensors for various applications. The steps are to produce NTD-Ge sensors first, then to study the dependence of their performances on the production parameters and finally to optimize their electric contacts.

In this paper we present the different possibilities for estimating and measuring the real neutron fluence received by each Ge wafer irradiated in a thermal neutron reactor. Measurements of their resistivity at 300K indicate a fluence discrepancy from the expected value and confirm the homogeneity of the doping throughout the volume. In addition we present a method allowing an improved estimation of the impedance below 30mK just by measuring the ratio of the NTDs' resistivity at 77K and 4K.

Keywords Thermal sensor • Cryogenic • Dark Matter • Double Beta Decay

1 Introduction

Although Metallic Magnetic Calorimeters (MMCs) and Transition Edge Sensors (TESs) have demonstrated the highest sensitivities and excellent energy resolutions, a certain number of experiments searching for Dark Matter (DM) or Neutrinoless Double Beta Decay (0νDBD) are using Neutron Transmutation Doped Germanium thermistors (NTDs) as the main solution for their heat channel measurements. Indeed in their present or even in their next stages, they take benefit from their robustness, reliability, ease of use over a large range of temperature and large dynamic range in energy. This is the case for the LUMINEU and EURECA projects, as examples.

The requirements for long term, large scale production and reproducibility and the need for improvements in energy resolution and threshold (for DM detection) have led us to develop NTDs with optimal and homogeneous doping in large volumes. Our development plan is first to demonstrate our ability to produce such sensors, then to study the dependence of their sensitivity on the production parameters and finally to realize them with optimal contacts and heat capacity.

2 Production of NTD-Ge for the LUMINEU experiment

2.1 Motivations

The development of thermal sensors in the LUMINEU project [1] is for two main applications. On one hand, it is to measure heat and light for scintillation-heat calorimeters used to search for neutrinoless double beta decay detection. On the other hand, it is for the heat measurement in ionization-heat detectors for DM WIMP searches.

Although NbSi meanders and MMC are under development within LUMINEU to improve the performance of the heat channel, NTDs are still the base line solution to satisfy the requirements of large projects such as CUPID (0vDBD) [2] and EURECA (DM) [3]. Present large scale experiments such as EDELWEISS and CUORE are equipped with NTDs provided by Lawrence Berkeley National Laboratory [4-5].

Below 1K, electric conduction in highly doped semiconductors, with impurity concentrations above 10^{15} cm^{-3}, is from tunneling between localized states (Variable Range Hopping). This depends strongly on the inter-dopant distance and therefore on the homogeneity of the dopant distribution. Among the different doping techniques, Neutron Transmutation Doping results in the best homogeneity [6].

2.2 Irradiation

Three inches 0.5 mm thick High Purity Ge single crystals (HPGe) have been introduced into the thermal neutron reactor Orphee at Saclay. Four wafers were irradiated at the same time, stacked together, facing the core of the reactor, without rotation. The thermal neutron flux was measured with a "collectron" at the same location just before the introduction of the wafers into the reactor, to estimate the irradiation time corresponding to the desired fluences. The variation of the neutron flux following the vertical axis over 60 cm was measured to be less than 1%. A further measurement was performed just afterwards to obtain the mean neutron flux during the irradiation.

Special care has been taken for the determination of the neutron fluences: 5mm long AlCo wires were joined to the wafers during the irradiation as dosimeters integrating the flux. Their activities have been measured with a standard HPGe detector at Laboratoire National Henri Becquerel (LNHB) in Saclay for a very precise measurement.

Table 1 presents for each irradiation the neutron fluence extracted from the collectron measurement and the time of irradiation, the expected activity of the AlCo dosimeter, the AlCo activities measured at LNHB and the neutron fluence required to get such activities. The discrepancy between the ^{60}Co expected and the 10%-15% lower measured activities is not understood. A simulation with GEANT4 has been conducted showing that the impact of the presence of the germanium wafers on the Co activation is in the order of 1% only. Since the dosimeters work as time integrators of the real neutron flux, in the following only the measured activity is considered as the reference.

The wafers were intentionally sub-irradiated to be sure not to overpass the optimal doping leaving room for a complementary irradiation, with a better control of the total fluence.

N°	Neutron fluence in 10^{18} n·cm^{-2}	Expected AlCo activity in Bq·mg^{-1}	Measured AlCo activity in Bq·mg^{-1}	Corrected neutron fluence in 10^{18} n·cm^{-2}
1	3.59	5685	4781	3.022
2	3.48	5520	4645	2.936
3	3.40	5377	4476	2.83
4	2.89	4571	No dosimeter	
5	3.69	5844	5155	3.26
6	3.56	5628	5065	3.20
7	3.46	5470	4884	3.085
8	3.29	5201	4752	3.005
9	3.08	4868	4370	2.76
10	2.91	4609	3969	2.51

Table 1 Neutron fluences extracted from the collectron measurement and the duration of irradiation, expected activity of the AlCo dosimeter at the end of the irradiation, AlCo activities measured at LNHB and corrected fluence according to the measured activity for each irradiation. AlCo (0.1% ^{59}Co) dosimeter masses have been normalized to 1mg. The error on the measured activity is in the range of 20 Bq·mg^{-1}.

2.3 Process of production

The metallization of the electrodes of our NTD thermal sensors did not follow the well-known procedure described by Haller et al. [6, 7]. Indeed, the boron ions were implanted first without annealing the crystals after the irradiation. The important point when metallizing is to get the metallic part as close as possible to the surface. It is also important that the implanted layer is completely amorphous for a better annealing. Otherwise residual crystal grains act as growing germ and the layer becomes polycrystalline. At CSNSM in Orsay, the energies of implantation of the metallic part of the electrode were chosen to be 5, 15 and 25 keV. These energies have been chosen to get a flat impurity profile [8]. To avoid any channeling, reduce the depth of implantation by 15 to 20% and get a more abrupt implanted ion profile, the crystals were tilted by 7° from the crystallographic axis.

Then Pd and Au layers of 10 nm and 800 nm thick respectively were evaporated under high vacuum at the technical platform Minerve of IEF in Orsay. It is only at this step of the production that the implanted ions have been activated by annealing at 350°C for 12 hours.

3 Resistivity measurements

3.1 4-point and 2-point measurements at very low temperature

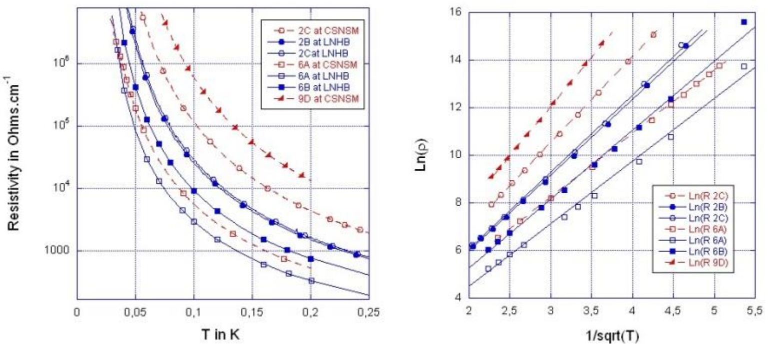


Fig. 1 *Left*: Resistivity ρ(*T*) curves for different NTD wafers and different irradiations (2, 6 and 9 correspond to 2.93, 3.2 and 2.76 $\cdot 10^{18}$n$\cdot$cm^{-2} resp.). Samples 2C, 6A and 9D (red dashed line) were measured at CSNSM in Orsay; 2B, 2C, 6A and 6B (blue solid line) at LNHB in Saclay. *Right*: same curves in ln(ρ) vs 1/sqrt(*T*) format. Errors on the resistivity measured with a Wheatstone bridge TRMC2 (Air Liquide) are lower than 1%. (Color figure online)

In order to validate the temperature dependence of the resistivity of the different wafers, NTD chips have been cut and measured in two dilution refrigerators. The dimensions of the chips are 4x4x0.45mm. Electrodes of 4x0.35mm^2 area are deposited along the two opposite edges of one of the 4x4 surfaces. Fig. 1 *Left* presents the R(T) curves of different NTD sensors. Fig. 1 *Right* presents the same curves in ln(R) vs 1/sqrt(T) format. The straight lines in the latter demonstrate that these NTDs follow perfectly the Efrös and Shklovskii law [9]: $\rho = \rho_0 \cdot \exp(\sqrt{T_0/T})$ (1)

As seen in Fig. 1 (left), the resistivity increases steeply upon cooling below 0.2 K, in fact so steeply that these sensors are useful as high-sensitivity devices in a narrow temperature range. The desired impedance depends on the temperature of the front end electronics: 1 to 10 MΩ for a FET at 110K. In this case, logarithmic sensitivities up to alpha=140 have been achieved for optimized operation, at 20 mK. Due to the intentional sub-irradiation (mentioned in part 2.2), the present sensors are only useable in the temperature range 40-90 mK. A complementary irradiation is required for use at lower temperature.

2.2 4-point measurements at 300K

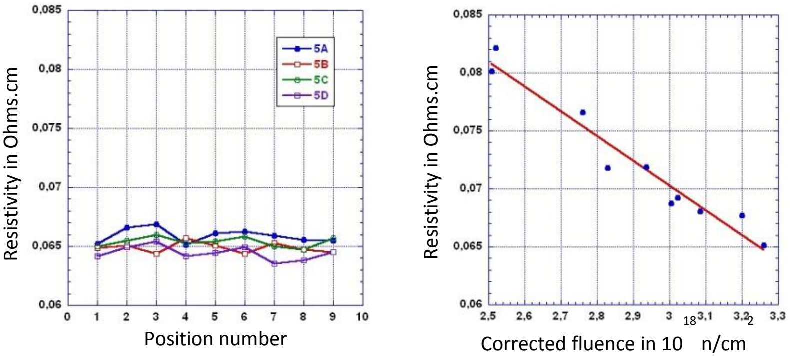


Fig. 2 *Left*: Resistivity measured at 9 different positions for each of the 4 wafers (A, B, C, and D) irradiated simultaneously in the 5th batch. Here the corrected fluence is 3.26 · 10^{18} n/cm^2. *Right*: Resistivity vs the fluence extracted from the cobalt dosimeters. (Color figure online.)

In order to study the homogeneity of the doping over the whole volume of a single wafer and between the wafers of the same irradiation, measurements of the resistivity at 9 different locations on the wafers were conducted with a 4-point tool at 300K, before the metallization and the cut of the wafers. This measurement took place at IAS Orsay at a temperature around 21°C.

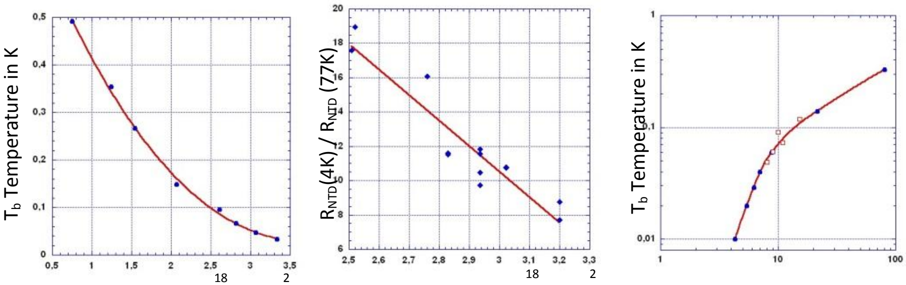
Results demonstrated that the resistivity is homogeneous over the whole surface of each wafer. Moreover they show that, to a good approximation, the wafers within an irradiation batch have the same doping. Fig. 2 *Left* shows a typical resistivity for the wafers of one of the irradiations. Fig. 2 *Right* presents the average resistivity vs the fluence extracted from the cobalt dosimeters. The behavior at very low temperature is strongly dependent on the doping (fig.1). The linear dependence (with a negative coefficient) of the resistivity at 300K relative to the fluence, in this range, demonstrates the possibility of estimating the resistivity of the NTDs at low temperature.

2.3 4-point measurements at 77K and 4K

The resistivity study described above has been repeated at 4.2K and 77K to get fixed temperature references. At this purpose, 3-inch-long bands were cut from the wafers and narrow strips perpendicular to their long axis spaced by 3.6 mm were metallized. Thus repetition of 4-point measurements was easily conducted to increase statistics. The most interesting parameter appeared to be the ratio *K* of the resistivity measured at 4K and 77K: we propose to use it as a new method of predicting the temperature at which the resistivity of the NTD is 10^5 Ω·cm, by only measuring the resistivity at 4K and 77K.

Fig. 3 *Left* shows the temperature T_b at which the resistivity ρ is 10^5 Ω·cm versus the fluence (extracted from Haller 94 [10]). Fig. 3 *Center* presents the *K* ratio relatively to the doping fluence. Fig. 3 *Right* gives the temperature T_b as a function of the *K* ratio. In the latter, data measured at IAS were obtained with NTDs of different doping and mounted on pure germanium pieces for thermalization without mechanical stress.

With such a predictive method, it seems realistic to determine the behavior at low temperature of the NTD sensors with a reduction of time and helium consumption.



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fluence. *Right*: Temperature T_b as a function of the K ratio. Blue points correspond to unpublished IAS data. Red points correspond to 5 NTD chips measured at CSNSM (see fig.1). (Color figure online.)

3 Conclusion

The results presented here demonstrate our ability to produce NTD thermistors. $R(T)$ curves confirm the quality of the electrical contacts and the homogeneity of the doping over the whole wafers. 4-point measurements at 300K, at 77K and 4K and in particular the ratio of resistivity measured at 4K and 77K show the possibility to get a good estimation of the resistivity at low temperature.

We are presently able to provide NTD sensors to experiments working between around 50mK and 100mK. Some wafers will be submitted to a complementary neutron irradiation for an optimization around 20mK.

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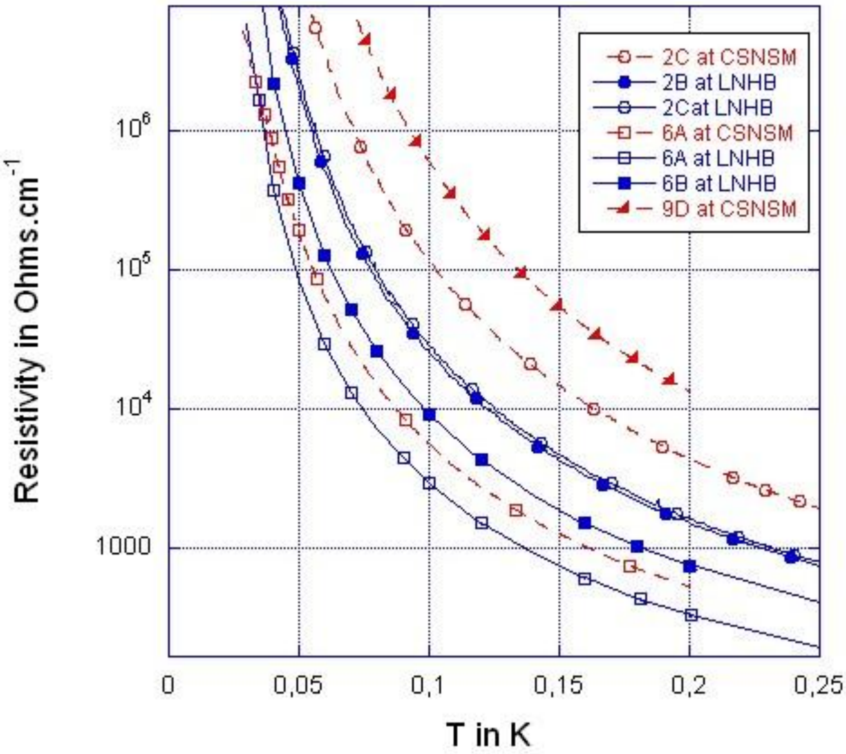

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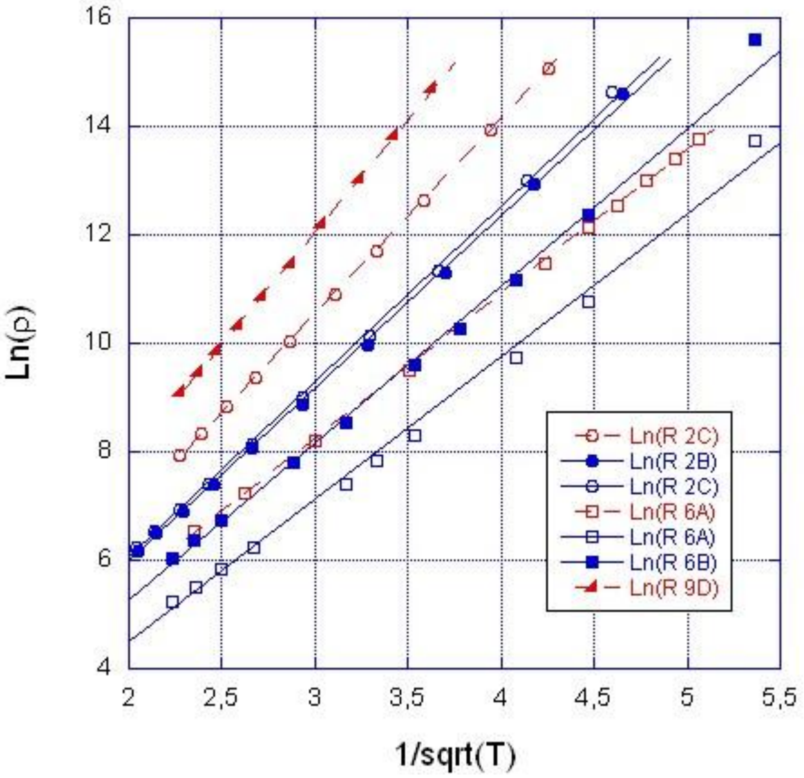

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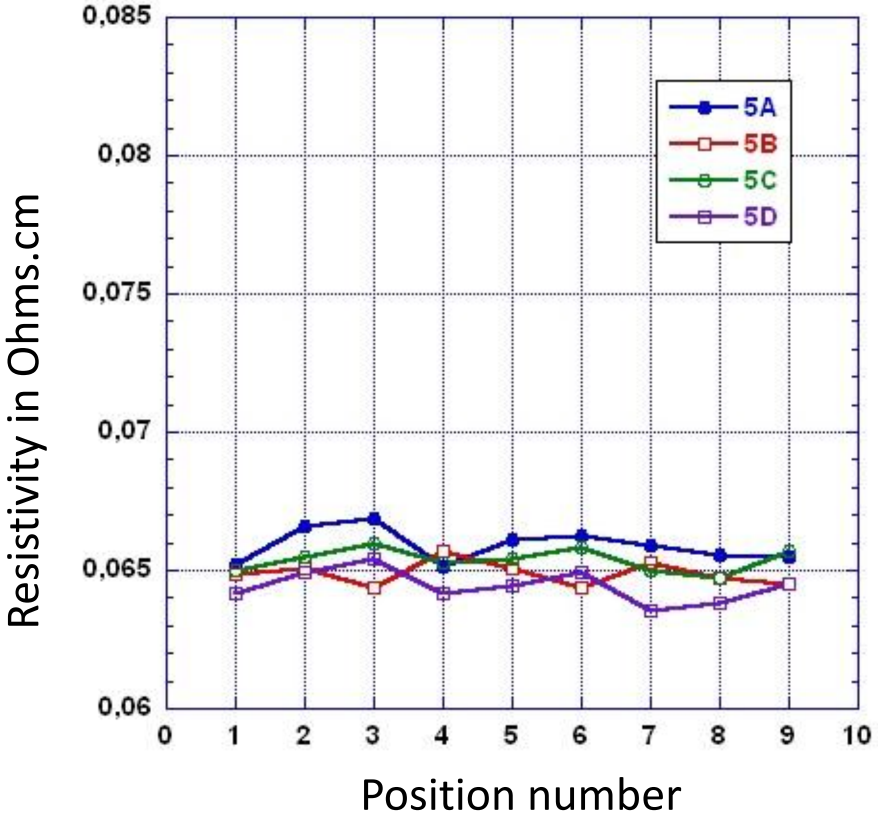

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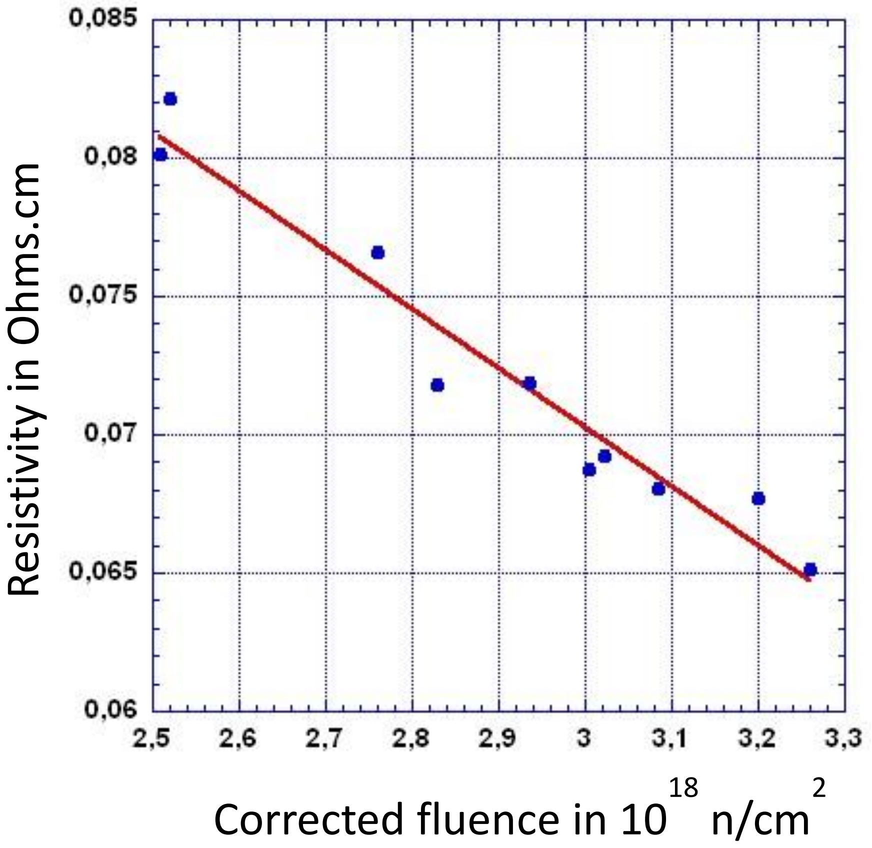

Fig. 2 *Right*: Resistivity vs the fluence extracted from the cobalt dosimeters. (Color figure online.)

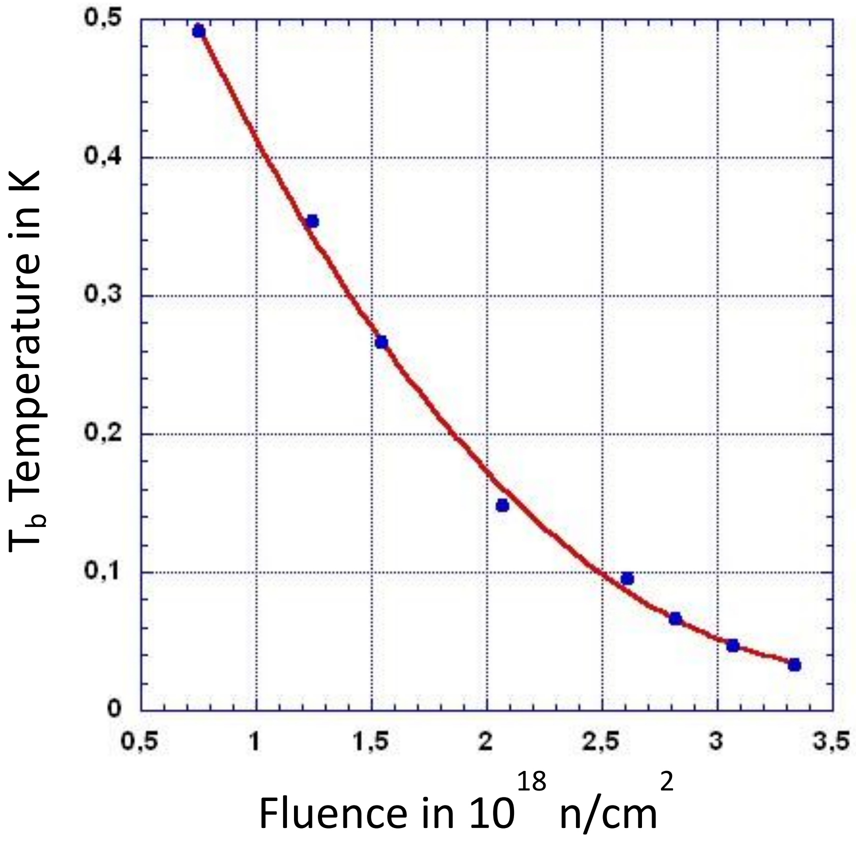

Fig. 3 *Left*: temperature T_b at which the resistivity ρ is 10^5 Ω·cm versus the fluence (extracted from Haller 94 [10]). (Color figure online.)

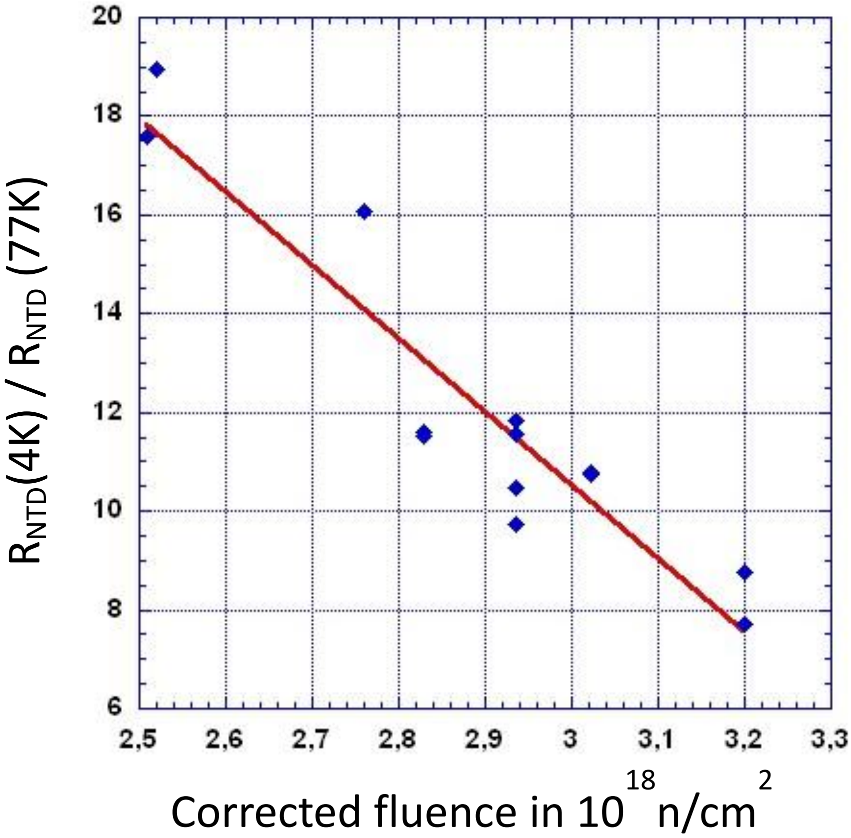

Fig. 3 *Center*: *K* ratio as a function of the fluence. (Color figure online.)

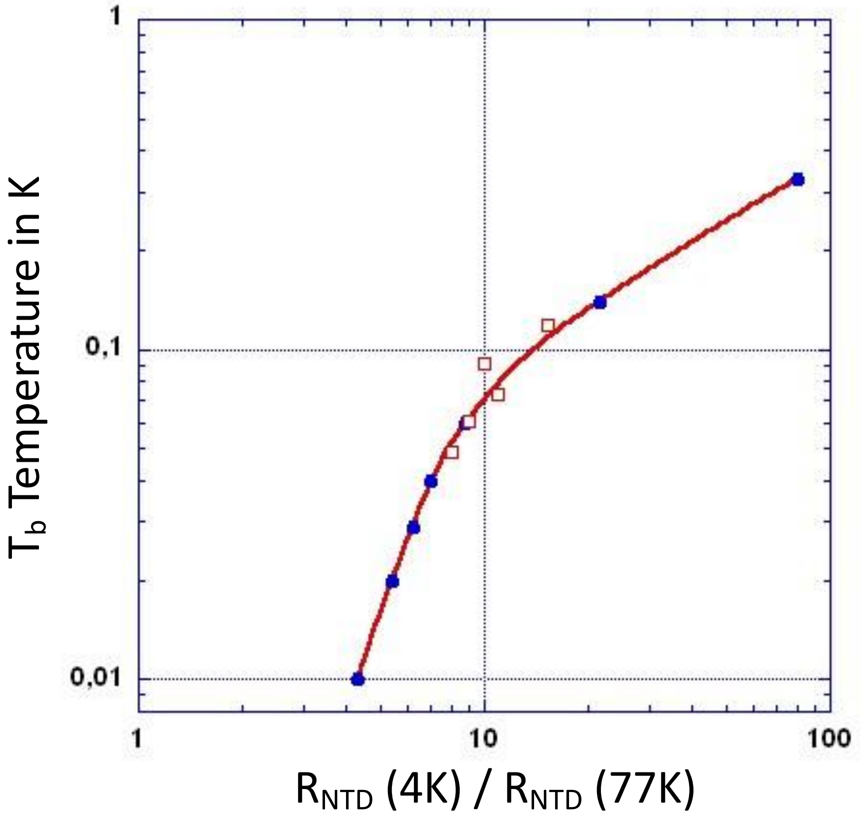

Fig. 3 *Right*: Temperature T_b as a function of the K ratio. Blue points correspond to unpublished IAS data. Red points correspond to 5 NTD chips measured at CSNSM (see fig.1). (Color figure online.)