DD

F. G. M. 100

HEAT CAPACITY OF LOW TEMPERATURE Ge- AND Si-CALORIMETERS AND
OPTIMIZATION OF As-IMPLANTED SILICON THERMISTORS

A. Alessandrello², D.V. Camin², G.F. Cerofolini³, E. Fiorini²,
A. Giuliani², C. Liguori², L. Meda³, T.O. Niinikoski¹
and A. Rijllart¹.

- 1) CERN, Geneva, Switzerland.
- 2) Dipartimento di Fisica dell'Universita' and I.N.F.N.,
Milano, Italy.
- 3) SGS Microelettronica, Agrate, Milano, Italy.

ABSTRACT

Low temperature calorimeters can be used to measure the total energy released in them by a single particle. Resolutions better than 1 eV are theoretically achievable with this kind of detectors. Preliminary experimental results in the range 80-1000 mK obtained with two different test detectors are reported. A reproducible process for the fabrication of high sensitivity thermistors is described. Future developments are briefly discussed.

CM-P00062962

1. INTRODUCTION

A crystal of a pure diamagnetic insulator can be used as a low temperature calorimeter in order to detect single particles [1-3]. The total energy deposited by the particle can be measured through the temperature increase of the whole mass of the crystal, with a resolution theoretically better than 1 eV. The changes of the lattice temperature are measurable by means of a resistance thermometer integrated in the crystal. This technique has been successfully used to detect and analyze X-rays[4,5] and alpha particles[6-8]. This new method looks very promising in several fields of physics: large mass calorimeters can detect rare nuclear and subnuclear events and exotic weakly interacting particles, also below the ionization limit[1,2,9,10]; small size detectors can be used in astrophysics[1-5,11,12] and for the measurement of the electron neutrino mass[2,7,13-16], where extremely good resolutions are required.

We discuss here some preliminary experimental work on test detectors, with the aim to gain insight in the physics of thermal-particle detection[8]. We have also determined a reproducible standard process for the construction of high sensitivity silicon thermistors[17]. This fabrication process is described and discussed.

2. TEST DETECTORS: EXPERIMENTAL DETAILS AND RESULTS

Two different calorimeters have been tested at various temperatures under 1 K.

The first calorimeter[8] was originally a Si:P resistance thermometer, provided us by G. Frossati[17], consisting of a wafer of n-silicon of $1 \times 0.5 \times 0.3 \text{ mm}^3$. A resistive area of $0.3 \times 0.5 \text{ mm}^2$ has been obtained by implanting phosphorus ions up to a depth of $1000 \text{ \AA}$ with a concentration of $1.5 \times 10^{19} \text{ atoms/cm}^3$. The electric contacts have been obtained by two $2 \text{ }\mu\text{m}$ degenerate n^+ implants of $10^{21} \text{ atoms/cm}^3$ and by $1 \text{ }\mu\text{m}$ thickness of aluminium deposited on them. The total volume of the implanted part is $6.15 \times 10^{-4} \text{ mm}^3$. Electrical and thermal contact to the detector was obtained with two indium wires of about $100 \text{ }\mu\text{m}$ diameter and 3 mm length, which are cold welded at one end to the aluminium contacts and at the other to a copper frame in thermal contact with the mixing chamber of a dilution refrigerator.

In order to characterize the detector sensitivity, we define an adimensional coefficient A as:

$$A = - \frac{d[\log R(T)]}{d(\log T)}$$

where $R(T)$ is the thermistor resistance and T the temperature. The value of A for the present detector ranges from 0.5 at 500 mK to 1.1 at 100 mK .

Pulses from the detector have been obtained by placing, inside the cryostat and in the immediate vicinity of it, an implanted ^{228}Ra source, in radiative equilibrium with its alpha-emitting daughters, which produces a counting rate of about 4 Hz . Measurements have been carried out at various temperatures from 30 to 960 mK , but in the lower range of this interval the

relative increases in temperature become so large that the response of the system is not linear. The present analysis has, therefore, been limited to measurements carried out at temperature above 300 mK.

Most of the information on the thermal properties of the calorimeter come from the pulse height and decay properties as a function of temperature. The decay pattern is compatible with the sum of two exponentials as expected from the thermal model of Fig. 1, where C_1 is the heat capacity of the silicon wafer, G_1 the heat boundary conductance between the implanted silicon and the aluminium-indium system, C_2 is the heat capacity of the aluminium layer and, mainly, of the indium contact, which must be taken into account because of the relatively large amount of indium required for the welding, and G_2 is the heat conductance of the indium wires to the copper frame, which acts as a heat sink.

The analysis of the pulse heights and shapes at the different temperatures allows us to determine C_1 , C_2 , G_1 and G_2 as a function of temperature. Particularly, experimental data[8] are in good agreement with the following functions for C_1 :

$$C_1 = a T^3 + b T$$

$$C_2 = a' T^3 + b'$$

where $a = (9.2 \pm 1.2) \times 10^{-11} \text{ J/K}^4$, $b = (1.5 \pm 0.4) \times 10^{-11} \text{ J/K}^2$, $a' = (10.3 \pm 0.9) \times 10^{-11} \text{ J/K}^4$ and $b' = (0.6 \pm 0.1) \times 10^{-11} \text{ J/K}$ [8]. The term which behaves as T^3 is due to the lattice contribution and the

term weakly dependent on temperature must be ascribed to the doped part. The values of a and a' are in excellent agreement with the value of 9×10^{-11} , predicted by a calculation based on the lattice vibration term. The values of G_1 and G_2 , which range respectively from 8×10^{-7} W/K to 269×10^{-7} W/K and from 12×10^{-7} W/K to 735×10^{-7} W/K [8] as temperature ranges from 363 mK to 958 mK, give total recovery times below few hundred microseconds. So the pulses are faster than those obtained in other experiments [4,5].

The present analysis [8] constitutes the first attempt to determine thermal properties and particularly the temperature dependence on heat capacities at low and ultralow temperatures, using the known energy delivered by an alpha particle. We think that this approach, which is of interest also for solid-state and low temperature physics, represents a necessary step towards the optimization of low temperature calorimeters as particle detectors.

The second calorimeter was the sensing element of a commercial germanium resistance thermometer, manufactured by Lake Shore Cryotronics. It consists of a germanium crystal of $3 \times 1 \times 0.7$ mm³ volume, melt doped with arsenic and gallium. Electrical and thermal contact to the calorimeter has been obtained with two gold wires of about 50 μ m diameter and 3 mm length.

The value of A for this thermistor ranges from 2 at 500 mK to 4.6 at 100 mK.

Pulses have been obtained with the same source and arrangement described for the silicon calorimeter. Measurements have been carried out at various temperatures, from 100 mK to 220

mK. Problems of temperature stability have prevented us from making measurements and analyses as accurate as those on the silicon calorimeter. However, we have roughly determined the calorimeter heat capacity as a function of temperature from the height of the pulses, as shown in Fig. 2. The experimental points are compatible with a T^3 behaviour, but the values of the heat capacity are about ten times higher than those expected from pure germanium lattice contribution.

Although detector resolution was not the aim of the present experiment, with the germanium calorimeter we have been able to separate the four main energy lines characteristic of the source.

3. THERMISTOR FABRICATION PROCESS

In order to improve the detector performance and to develop calorimeters suitable for specific applications, it is very important to establish a reproducible process for the construction of high sensitivity thermistors. For this reason one of the first aims of this work was the determination of the n^+ dopant concentration in silicon with optimum resistivity and sensitivity[16]. Preliminarily, we chose arsenic as n^+ dopant for two reasons: absence of the magnetic contribution to the specific heat, and a wellknown reconstructible ion-implanted layer.

The thermistors were prepared in Central Research and Development of SGS Microelettronica (Agrate, Milano, Italy) as described in the following flow-chart, where all steps are fully compatible with standard silicon planar technology:

- 1) Substrate: P-type, (100) oriented, 16-24 ohm·cm.

- 2) Ion implant: B^+ , $E=40$ keV, $\Phi=5 \times 10^{11} \text{ cm}^{-2}$.
- 3) Oxidation: dry O_2 , 1000°C , 180 min.; $1000 \text{ \AA}$.
- 4) Diffusion: N_2 , 1100°C , 120 min.
- 5) SiO_2 deposition: by oxidation of SiH_4 ; $5000 \text{ \AA}$.
- 6) 1st mask: defines n^{++} regions.
- 7) n^{++} ion implant: As^{++} , $E=150$ keV, $\Phi=6 \times 10^{15} \text{ cm}^{-2}$.
- 8) Annealing: N_2 , 550°C , 2 h.
- 9) Oxidation: steam, 1000°C , 15 min.; $2200 \text{ \AA}$.
- 10) Diffusion: N_2 , 1100°C , 200 min.
- 11) SiO_2 deposition: by oxidation of SiH_4 ; $4000 \text{ \AA}$.
- 12) 2nd mask: defines thermistor.
- 13) n^+ ion implant: As^+ .
 - 1st step: $E=50$ keV, $\Phi=\Phi_0$.
 - 2nd step: $E=100$ keV, $\Phi=\Phi_0$.
 - 3rd step: $E=150$ keV, $\Phi=2\Phi_0$.
 - $5 \times 10^{12} \text{ cm}^{-2} < \Phi_0 < 3 \times 10^{14} \text{ cm}^{-2}$.
- 14) Diffusion/oxidation: N_2/O_2 , 950°C , 30 min.; $200 \text{ \AA}$.
- 15) 3rd mask: defines contacts.
- 16) Al:Si (1%) evaporation; $1 \mu\text{m}$.
- 17) 4th mask: defines metals.
- 18) Al-Si alloy, H_2 , 450°C , 30 min.

The three step implant was chosen to have an almost flat in-depth concentration profile. The profile at the end of the process is shown in Fig. 3.

A lot of samples with different concentrations, in the range $0.2 \times 10^{19} - 11 \times 10^{19} \text{ atoms/cm}^3$ were prepared and characterized at temperatures under 1 K. The results show that an acceptable

combination of sheet resistivity ($\approx 4\text{K}\Omega/\square$) and sensitivity ($A = 4.8$) at the temperature of 0.1 K is obtained in a very narrow concentration range centred on 7.6×10^{18} atoms/cm³. An example of resistance-temperature characteristic is shown in Fig. 4.

The dimension of the sample used in this characterization were quite large, of about $15 \times 15 \times 0.5$ mm³; this size was chosen not to have problems associated with mechanical handling, electrical and thermal contacting and so on. In spite of this, one of the samples was bombarded with alpha particles. Pulses have been obtained at temperatures under 200 mK.

4. FUTURE DEVELOPMENTS

For the moment, our experimental work has been aimed to determine the thermal properties of the calorimeters and to study the mechanism of production and evolution of the thermal pulses. In the future, our search will continue into two directions.

The first aim is the realization of small size, high resolution detectors. For this purpose SGS is preparing Si:As calorimeters according to the reported flow chart, with chip dimension of $0.5 \times 0.5 \times 0.5$ mm³ and with a resistance-temperature characteristic similar to the one shown in Fig. 4.

The second aim is the development of relatively large γ -detectors, consisting of pure germanium or silicon crystals.

REFERENCES

- [1] T.O. Niinikoski and F. Udo: "Cryogenic Detection of Neutrinos", NP internal report 74-6, 1974 (Available from CERN Scientific Information Service, CH-1211 Geneve 23, Switzerland).
- [2] E. Fiorini and T.O. Niinikoski: Nucl. Instr. and Methods, 224, 83(1984), also for previous references.
- [3] S.H. Moseley, R.L. Kelley and D. McCammon: J. Appl; Phys., 56, 1257 (1984).
- [4] D. McCammon, S.H. Moseley, J.C. Mather and R.F. Mushotzky, J. Appl. Phys., 58, 1263(1984).
- [5] S.H. Moseley, R.L. Kelley, J.C. Mather, R.F. Mushotzky, A.E. Szymkowski and D. McCammon: to be published on IEEE Trans. Nucl. Sci.
- [6] C. Boragno, U. Valbusa, G. Gallinaro, D. Bassi, S. Iannotta and F. Mori: Cryogenics, 24, 681(1984).
- [7] N. Coron, G. Dambier, G.J. Focker, P.G. Hansen, G. Jegoudez, B. Jo, J. Leblanc, J.P. Moalic, H.L. Ravn, H.H. Stroke and O. Testard: Nature, 75, 314(1985).
- [8] T.O. Niinikoski, A. Rijllart, A. Alessandrello, E. Fiorini and A. Giuliani: Europhysics Lett., 1(10), 499(1986).
- [9] B. Cabrera, L.M. Krauss and F. Wilzek: Phys. Rev. Lett., 55, 25(1985).
- [10] A.K. Drukier: "Ultralow Background Detectors for Weakly Interacting Particles", presented at "3rd Pisa Meeting on Advanced Detectors", Castiglione della Pescaia (Italy) June

2-7, 1986.

- [11] P.M. Downey, A.D. Jeffries, S.S. Meyer, R. Weiss, F.J. Bachner, J.P. Donnelly, W.T. Lindney, R.W. Mountain and D.J. Silversmith: Appl. Optics, 23, 910(1984).
- [12] I.G. Nolt, J.V. Radostiz, M. Carlotti, B. Carli, F. Mencaglia and A. Bonetti: Int. J. of Infrared and Millimeter Waves, 6, 707(1985).
- [13] D. McCammon, M. Juda, D.D. Reeder, R.L. Kelley, S.H. Moseley and A.E. Szimkowiak: "A New Technique for Neutrino Mass Measurement", preprint.
- [14] A. Blasi, G. Gallinaro, B. Osculati, S. Sini, U. Valbusa and S. Vitale: "Una Nuova Determinazione di Limiti di Massa per l'Antineutrino", preprint.
- [15] Milano Group: "Proposal for the Thermal Determination of the Neutrino Mass", preprint.
- [16] G.F. Cerofolini, R. Bez and L. Meda: "Low Temperature Silicon Calorimeters as Detectors of Rare Subnuclear Events", preprint.
- [17] G. Frossati, D. Imbert, L. Peccoud, D. Thouluze and B. Wajsman: "Ion Implanted Silicon Thermometers for Very Low Temperatures", presented at "Low Temperatures Physics LT 14", Helsinki, August 1975.

FIGURE CAPTIONS

Fig. 1 - Thermal model for the silicon calorimeter.

Fig. 2 - Heat capacity versus temperature for the
germanium calorimeter.

Fig. 3 - Concentration profile for the Si:As thermistors.

Fig. 4 - Resistance-temperature characteristic for
a Si:As thermistor.

Fig. 1

Fig. 2

Fig.3

Fig. 4