Universitá degli Studi di Genova
Facoltá di Scienze Matematiche, Fisiche e Naturali

Anno Accademico 2004/05

Tesi di Dottorato di Ricerca in Fisica

Sviluppo di rivelatori criogenici microcalorimetrici per decadimenti beta a bassa energia

Development of cryogenics detectors for low energy beta decays

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Introduction

Low temperature micro-calorimeters particles detectors are going to play a leading role in many fundamental and applied physic disciplines. The work of my thesis deals with the nuclear and particle physics applications of these detectors. Above all, the main aim, which motivated my work, is the development of low temperature detectors for the direct neutrino mass measurement; this study is executed within the project MANU2 of INFN (Nuclear Physics National Institute) which aims to set a neutrino mass upper limit by the analysis of the $^{187}$Re beta decay whose end point energy is about 2.5 keV.

Whether these detectors could probe the indicated mass region around 0.1 eV/$c^2$ or not, is a crucial issue that must be addressed with investigations on the microcalorimeter physics and technology.

In spite of the good energy resolution predictions of micro-calorimeter models, the present performances are far from the ones needed to design a neutrino mass experiment with the sensitivity stated before. Therefore a detailed analysis of the sensors has been carried out in order to improve their performances.

Another important aim, that has been achieved in this work, is the possibility of make superconducting energy absorber in which the radioactive source is homogeneously dispersed inside. Inner source absorbers permit precise calorimetric measurements especially useful for the study of low energy nuclear physics such as $^{187}$Re (beta decay, $Q=2.5$ KeV), $^{163}$Ho (electron capture, $Q=2.8$ KeV), $^7$Be (electron capture, emitting 110 eV), and others. Because of the extremely low energy released in these decays it is very difficult to have accurate measurements of their spectrum with conventional detectors. Moreover even the possibility of refining nuclear and atomic energy spectra is enhanced by the use of superconducting absorber prepared with this technique.

In the first chapter an overview of neutrino physics is presented particularly for the direct measurements of neutrino mass.

The second chapter deals with the low temperature Transition Edge Sensor (TES) based microcalorimeters physics and with the problem of the
excess noise in TES and the possible mechanism of generation.
The third chapter reports on the analysis of thermal sensors suitable for microcalorimeter to be used in the MANU2 experiment with superconducting rhenium absorber. A first part is dedicated to the Ag-Al TESs which have shown contradictory performances. Therefore the chemical-physical properties have been investigated to better understand the role of interdiffusion and the chemical activity of the two metals. In conclusion the use of stable Ag-Al alloy for TES superconducting film is proposed and tested. Other systems have been considered for TES: the IrAu and the dilute alloy of Al:Mn and Zn:Mn. At the end, I will describe the magnetic Au:Er sensors developed by the Heidelberg group, not only for the promising result obtained as x-ray detectors, but mainly as a real implementation for the $^{187}$Re experiment.

In the fourth chapter I move the attention on the microcalorimeter absorber and I will discuss the general features of the absorber materials. Following the topic of the forth chapter, in the fifth I will describe the possibility to have absorbers with the radioactive source homogeneously dispersed inside. In particular this work was finalized to prepare a superconducting absorber for a full energy absorption spectroscopy of $^{163}$Ho electron capture decay. A suitable absorber has been selected for a measurement with high energy resolution.

In the sixth chapter the last results regarding MANU2 sensors development are presented, in particular the good result obtained with IrAu TES which lead to an energy resolution of 5.9 eV on the Kα line with the consequent identification of Kα1 and Kα2 lines. The last chapter looks to the future, it deals with my work done in collaboration with the low temperature detector group of Heidelberg in order to study the possibility to use magnetic Au:Er sensor in experiments where the energy absorber is a superconductor as rhenium.

The following scheme will summarize the work of my thesis and above all I wanted to show which are the parts leading new contributions.
Figure 1: Simple structure of the thesis
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Chapter 1

Neutrino physics

1.1 Introduction

The existence of a particle called neutrino was first proposed by Pauli in 1930 [1] while a complete theoretical formulation, in agreement with the experimental results, has been advanced by Fermi [2] in 1934 (considering a zero mass particle). It became immediately clear that neutrinos would be difficult to observe because the smallness of their interaction cross-sections. The question whether neutrinos are massive particles has been a fundamental problem over 70 years.

The number of neutrinos which take part in weak interactions has been determined by the precise measurement of the decay width of Z boson and it has been found to be 3, (2.994 ± 0.012 from the combined fit to all LEP data [3]). Neutrino masses have been investigated through the endpoint analysis of β decay spectra in which a neutron inside the nucleus decays emitting one electron and one electron anti neutrino, following:

\[ N \rightarrow P + e^- + \bar{\nu}_e \]  

(1.1)

In low energy decay, almost the total available energy is shared between the neutrino and the electron, while a small part goes in nuclear or atomic excited states. Measuring the electrons energy spectrum the sensitivity to electron neutrino mass \( m_{\nu_e} \) comes from the relation:

\[ \frac{dN}{dE} \propto \sqrt{(Q - E)^2 - m_{\nu_e}^2} \]  

(1.2)

where Q is the maximum energy available for the decay. These experiments succeeded only in placing increasingly more restrictive upper limits. The best electronic neutrino mass upper limit was set by the experiments of Troisk and Mainz studying the tritium β spectrum: they found
\( \nu_e < 2.2 \text{ eV} \) at 95\% C.L.\cite{4}.

An important evidence that neutrinos are massive particles comes only from recent indirect measurements on neutrino flavor appearance/disappearance. In 1968 Gribov and Pontecorvo suggested that a neutrino of a given initial flavor could interchange its identity with other flavors, with a probability that is dependent on the neutrino mass eigenstates and mixing angles, and on the distance from the location of the source\cite{5}.

This theory was considered to explain why experiments measuring the solar neutrino flux showed a deficit in counts compared to the Standard Solar Model (SSM) expectations. The solar nuclear reactions provide a huge electron neutrino flux (their energy spectrum is showed in Fig. 1.1) as calculated in the framework of SSM.

When the \( ^{37}\text{Cl} \) radiochemical experiment of Davis\cite{6}, with an energy threshold of 750 keV and therefore able to detect neutrinos from the \( ^7\text{Be} \) line, began to show a deficit in the number of detected neutrinos, several questions arose in the scientific community. This deficit was also
measured by the $^{71}$Ga radiochemical experiment: SAGE [7], GALLEX [8], GNO [9] which had a low enough energy threshold to detect neutrinos from the pp cycle.

The first convincing evidence of neutrino oscillations was only recently reached in 1998, after the Super Kamiokande (SuperK) measurement [10] of atmospheric neutrinos. Atmospheric neutrinos are produced by high energy cosmic rays colliding with atoms in the upper part of the atmosphere and thus giving life to pions and kaons showers and therefore to their decays:

$$\pi^+ K^+ \rightarrow \nu_\mu \mu^+ \rightarrow \nu_\mu e^+ \nu_e \bar{\nu}_\mu$$  \hspace{1cm} (1.3)

$$\pi^- K^- \rightarrow \bar{\nu}_\mu \mu^- \rightarrow \bar{\nu}_\mu e^- \nu_e \nu_\mu$$  \hspace{1cm} (1.4)

From these relations the ratio between the electron neutrinos and muon neutrinos is 1/2 and they are characterized by energy ranging in the GeV region. SK measurements demonstrated that the muon neutrino flux had a deficit, if compared to the theoretical expectations, which is related with the zenith angle, while the electron neutrino flux was consistent with the expected value.

After this former result, experiments measuring solar and atmospheric neutrino fluxes consolidated the neutrino oscillations hypothesis setting important limits on mixing angles and square mass differences. Among them it is important to remember the contribution given by SNO (Sudbury Neutrino Observatory) that with the possibility to measure both charge current and neutral current neutrino interactions, which strongly reduced the allowed parameters regions [11].

Neutrino properties are investigated by other type of appearance/disappearance experiment measuring neutrinos from reactor or accelerator. Nuclear reactor are very intense source of $\bar{\nu}_e$ from the beta decays of the neutron rich fission fragments. Knowing precisely the reactor neutrino spectrum and the neutrino cross section, the number of events in the detector may be estimated (see fig. 1.2).

Oscillation search using reactors as sources are very interesting since they can dispose of a 'known' neutrino flux of energy of few MeV and the well determined distances ranging from about 1 Km as CHOOZ and Palo Verde to 100 km for KamLAND.

There is another man-made neutrino source: the accelerator neutrino whose energy spectrum in the GeV range can be well known. These neutrinos come from the interaction of high energy protons beam with a target and the following production of pions and kaons. The experiments designed for accelerator neutrinos are MINOS at Soudan mine looking for neutrinos coming from FNAL and the analogous projects between
Figure 1.2: Reactor neutrino energy spectrum, neutrino cross-section and expected interactions

CERN and Gran Sasso, OPERA and ICARUS. These two kind of measurement will provide information on the $\theta_{13}$ and on the CP-violating phase.

It is evident how many have been developed to investigate neutrino oscillations and even if they can determine different neutrino properties, they are not dependent on the absolute neutrino mass. Therefore it seem utmost important to develop experiments able to directly measure the neutrino mass. At the moment the most accredited experiment is KATRIN on the analysis of the $^3H$ beta spectrum, but the MANU2 experiment in which the $^{187}Re$ beta spectrum is studied will be able to reach the sensitivity of 2 eV/$c^2$ on neutrino mass before the KATRIN taking data and therefore be an interesting comparison with tritium future data.

In the next section I will give an overview on neutrino properties on the basis of recent result, then I will describe the methods to directly measure their mass, task becoming more and more important at the light of
the new neutrino oscillations parameters.

1.2 Neutrino properties and recent results

All the experimental results are consistent within the framework of two different sets of three neutrino flavors and three mass eigenstates, therefore each flavor eigenstate $\nu_\alpha$ ($\alpha = e, \mu, \tau$), may been written as a superposition of the three mass eigenstate $\nu_i$ ($i=1, 2, 3$) through an unitary matrix $V$:

$$\nu_\alpha = \sum V_{\alpha i}^* \nu_i \quad (1.5)$$

The mixing matrix $V$ is specified by three rotation angles $\theta_1, \theta_2, \theta_3$ ($0 < \theta_i < \pi/2$) and three CP-violating phases $\delta, \phi_2$ and $\phi_3$ ($0 \leq \delta, \phi_i \leq 2\pi$). $V$ can be written as:

$$V = \begin{bmatrix}
1 & 0 & 0 \\
0 & c_{13} & s_{13} \\
0 & -s_{13} & c_{13}
\end{bmatrix} \begin{bmatrix}
c_{23} & 0 & s_{23} e^{-i\delta} \\
0 & 1 & 0 \\
-s_{23} e^{i\delta} & 0 & c_{23}
\end{bmatrix} \begin{bmatrix}
c_{12} & s_{12} & 0 \\
-s_{12} & c_{12} & 0 \\
0 & 0 & 1
\end{bmatrix} \quad (1.6)$$

$$\begin{bmatrix}
1 & 0 & 0 \\
0 & e^{i\frac{\phi_2}{2}} & 0 \\
0 & 0 & e^{i(\frac{\phi_3}{2}+\delta)}
\end{bmatrix} \quad (1.7)$$

where $c_i$ denotes $\cos \theta_i$, $s_i$ denotes $\sin \theta_i$. Therefore the three flavors neutrino oscillations are parameterized in term of the three neutrino masses ($m_1, m_2, m_3$) and of the three mixing angles, and the CP-violating phase. The oscillation probabilities are independent of the Majorana phases $\phi_2$ and $\phi_3$. Vacuum neutrino oscillations are given by:

$$P(\nu_\alpha \rightarrow \nu_\beta) = |\sum_j V_{\alpha j} e^{-\frac{i m_j^2 L}{2 E_{\nu_j}}} V_{\beta j}^*|^2 \quad (1.8)$$

The oscillation probabilities depend on the square-mass difference, the mixing angle and on the distances. If matter effects are considered, then the oscillation probabilities would take into account the electron number density of the interposed medium.

The present experiments measuring solar and atmospheric neutrino fluxes set two independent $\Delta m^2$ and of different order of magnitude, $\Delta m^2_{SUN} << \Delta m^2_{ATM}$, the first identified with the oscillations of electron neutrino and therefore $\Delta m^2_{SUN} = \Delta m^2_{12}$ while the second is related to the oscillation of $\nu_\mu$ to $\nu_\tau$, $\Delta m^2_{ATM} = |\Delta m^2_{31}|$. Since the sign of $\Delta m^2_{31}$ is not known, two different schemes are allowed by the hierarchy $\Delta m^2_{SUN} << \Delta m^2_{ATM}$,
the normal scheme $m_1 < m_2 << m_3$ in which $\Delta m^2_{21}$ is positive and the inverted scheme $m_3 << m_1 < m_2$ with $\Delta m^2_{31}$ negative.

Fogli and al. [12] made a global fit of available data on neutrino physics properties, Giunti [13] reported a table with the value of atmospheric and solar parameter (tab. 1.1).

The three mass states can be written in function of the two squared mass differences as:

$$ (m_1^2, m_2^2, m_3^2) = \mu^2 + \left( -\frac{\Delta m^2_{12}}{2}, +\frac{\Delta m^2_{12}}{2}, \pm \Delta m^2_{31} \right)$$

(1.9)

where $\mu$ fixes the absolute neutrino mass scale and the sign of $\Delta m_{31}$ identified the normal or inverted hierarchies.

Even if many important experiment are investigating with high accuracy neutrino oscillations parameters, it is now extremely interesting to measure the absolute mass scale, therefore direct measurements of neutrino masses are being considered as fundamental importance.

In the next section I briefly describe the direct neutrino mass measurement, focusing on the beta spectrum analysis.
<table>
<thead>
<tr>
<th>Parameter</th>
<th>Best fit</th>
<th>3σ range</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\Delta m^2_{21}$</td>
<td>$8.3 \times 10^{-5} eV^2$</td>
<td>$7.4 \times 10^{-5} - 9.3 \times 10^{-5} eV^2$</td>
</tr>
<tr>
<td>$\sin^2 \theta_{12}$</td>
<td>0.28</td>
<td>0.22-0.37</td>
</tr>
<tr>
<td>$</td>
<td>\Delta m^2_{31}</td>
<td>$</td>
</tr>
<tr>
<td>$\sin^2 \theta_{31}$</td>
<td>0.01</td>
<td>0-0.05</td>
</tr>
<tr>
<td>$\sin^2 \theta_{23}$</td>
<td>0.5</td>
<td>0.32-0.68</td>
</tr>
</tbody>
</table>

Table 1.1: Best fit and 3σ range for the three neutrino oscillations parameters

1.3 Direct search for neutrino mass

Measurement of neutrino masses are extremely complicated (both for the set up and the data analysis) due to the smallness of the effects they want to see. Three main groups of experiment may be considered: cosmological and astrophysical measurement, neutrinoless double beta decay and the analysis of the and point of low energy beta decay ($^3H$ and $^{187}Re$).

In the first group the analysis of Cosmic Microwaves Background are considered (CMB) and the Supernova Neutrino. In the first case massive neutrino play a role in the suppression of density fluctuations in the early Universe at small scale of order of 1 10 Mpc. The suppression depends on the sum of neutrino masses $\sum_k m_k$. Recent measurements of CMB by WMAP [14] and in the Large Scale Structure of galaxies, with other kind of measurements, set a stringent upper limit [13]:

$$\sum_k m_k < 0.5 eV$$  \hspace{1cm} (1.10)

If the Lyman-α forest is considered (even if still controversial) the same limit is lower:

$$\sum_k m_k < 0.1 eV$$  \hspace{1cm} (1.11)

Another important constrain for neutrino mass comes from neutrino emitted during a supernova explosion, neutrino mass information come from the measurement of the arrival time distribution. The current
limit was obtained detecting neutrinos coming from SN1987 and it is set around 23 eV [15]. Systematic uncertainties connected with not precisely known neutrino emission time spectrum doesn’t allow a sub-eV sensitivity.

Neutrinoless double beta decay \( (2\beta0\nu) \) can be written as:

\[
N(A, Z) \longrightarrow N(A, Z + 2) + e^- + e^-.
\]  

(1.12)

This kind of processes are forbidden in the Standard Model since there is a change of two units in the total lepton number. \( 2\beta0\nu \) is a very important process since it is not only sensitive to a non zero neutrino mass, but mainly because it is allowed only if neutrinos are Majorana particles. The corresponding half life is:

\[
T_{1/2}^{0\nu} = \left( G_{0\nu} |M_{0\nu}|^2 |m_{\beta\beta}|^2 \right)^{-1}
\]  

(1.13)

where \( G_{0\nu} \) is the phase space factor, \( M_{0\nu} \) is the nuclear matrix element and

\[
m_{\beta\beta} = \sum_k V_{ik}^2 m_k = |c_{13}^2 c_{12}^2 m_1 + c_{13}^2 s_{12}^2 m_2 e^{i\phi_2 + s_{13}^2 m_3 e^{i\phi_3}}|.
\]  

(1.14)

where \( \phi_2 \) and \( \phi_3 \) parameterize relative and unknown Majorana phases. If \( 2\beta0\nu \) events happens, the experimental sign is an isolated peak at a precise energy above the \( 2\beta2\nu \) continuum spectrum.

Up to now neutrinoless double beta decay experiments place only upper bounds on \( m_{\beta\beta} \), the only exception is the Heidelberg-Moscow [16] experiment which claims a positive signal from the analysis of \( ^{76}\text{Ge} \) isotope. From this signal they extrapolated the decay half life \( T_{1/2}^{0\nu} \) expressed in years, within the following 3\( \sigma \) interval:

\[
\log_{10}(T_{1/2}^{0\nu}/y) = 25.08^{+0.54}_{-0.24}
\]  

(1.15)

The half life permits to calculate the Majorana neutrino mass using:

\[
m_{\beta\beta}^2 = \frac{m_e^2}{C_{mm} T}
\]  

(1.16)

where \( m_e \) is the electron mass and \( C_{mm} \) is the nuclear matrix element for that isotope, but the knowledge of that is full of uncertainties and therefore systematic errors are unknown. From the global analysis in [12] the best estimation is:

\[
\log_{10}(m_{\beta\beta}/eV) = -0.23 \pm 0.18 \ (2\beta0\nu \ signal \ assumed),
\]  

(1.17)
The third group of experiments and the most promising for the determination of neutrino mass, is the analysis of the endpoint of beta decay spectra. The process investigated is of type \( N(A, Z) \rightarrow N(A, Z + 1) + e^- + \bar{\nu}_e \), a three bodies decay in which the electrons energy spectrum is measured. From this latter the Kurie plot, \( K(E) \), is calculated:

\[
K^2(E) = (Q - E) \sum_k |V_{ek}|^2 \sqrt{(Q - E)^2 - m_k^2}
\]

the sensitivity to a non zero neutrino mass is stronger when \((Q-E) \sim m_k\). Since the present experiments can not see any effect on neutrino masses \( m_k \), it is possible to approximate \( m_k << (Q - E) \) and obtaining:

\[
K^2(E) \simeq (Q - E)\sqrt{(Q - E)^2 - m_\beta^2}
\]

This function depends on only one parameter the effective neutrino mass:

\[
m_\beta^2 = \sum_k |V_{ek}|^2 m_k^2 = c_{12}^2 c_{13}^2 m_1^2 + s_{12}^2 c_{13}^2 m_2^2 + s_{13}^2 m_3^2
\]

Since the number of counts in the end point region is proportional to \((E/Q)^3\), to have a higher statistic, it is necessary to use isotopes which have the lower endpoint energy as possible. At the moment two are the decays used to neutrino mass study, the superallowed transition of \(^3\)H with endpoint energy of 18.6 keV and half life of 12.3 years and forbidden transition of \(^{187}\)Re at 2.46 keV but with a half life of order then \(10^{10}\) years.

The best result has been obtained by the Mainz and Troitsk tritium experiment and the upper limit set at \(\beta < 2.2 \text{ eV} \) at 95% CL. In the near future KATRIN a new upgraded tritium experiment will reach a sensitivity to neutrino mass down to \(1 \times 10^{-2} \text{ eV}\).

\(^{187}\)Re beta spectrum is at the moment studied by two experiments using cryogenic detectors: MANU2 by the group of Genoa and the Neutrino Mass Experiment by the group of Milan.

In the following sections I will describe the cryogenic detectors contributions to the neutrino physics and focusing the attention on the rhenium experiment performed by the group of Genoa.

### 1.4 Neutrino physics and low temperature detectors

Cryogenic detectors play a significative role in neutrino physics investigation, in fact they are used in two kind of experiments: in double beta
decay such as CUORICINO (CUORE) [17] and in experiment for direct measurement of neutrino mass studying $^{187}$Re beta decay. In particular for low energy beta decay such as the rhenium decay, cryogenic microcalorimeters are the more promising devices to reach the suitable energy resolution to set a neutrino mass upper limit competitive with the one obtained with the analysis of the tritium beta spectrum. The next paragraph describes the feature of the rhenium experiment and I will conclude with the description of MANU experiment.

1.4.1 The 187-Re experiment

The beta decay of $^{187}$Re has been observed in 1965 and in 1967 by means of proportional counter. In 1985 independently Dan McCammon [19] and Sandro Vitale [18] proposed the use of microcalorimeters for determining the neutrino mass. In the same year Sandro Vitale argued that the feasibility of such kind of measurement would be much more sensitive by using the 187Re, due to the lowest Q value known in nature. The simplest physical form in which a $^{187}$Re is available is the metal, that is superconducting below 1.69 K. In 1990 the Genoa Group found that metallic sample of rhenium is suitable for microcalorimeter applications. Only in 1992 the Genoa group detected the $^{187}$Re beta decay by means of cryogenic microcalorimeter with a poly-crystalline absorber of metallic superconducting rhenium.

In fact the reason for this late determination of the spectrum is also the reason for its importance in the measurement of electron neutrino mass upper value, the low end point energy, about 2.5 keV. Despite of the lower endpoint energy and the fact that radioactive $^{187}$Re represent the 62.6 % of natural rhenium, the measurement of its spectrum is complicated by the extremely long half life, calculated to be $4.12 \times 10^{18}$ years equivalent to $1.4 \times 10^{18}$ seconds.

The $^{187}$Re decay in $^{187}$Os following the relation:

$$^{187}\text{Re} \rightarrow ^{187}\text{Os} + e^- + \bar{\nu}_e$$

(1.22)

The shape of the electron energy spectrum is given by:

$$N(E_e, Z, m_\nu) = \sum_i \pi_i F(Z, E_e) S(E) p_e E_e (Q_i - E_e)^2 \sqrt{1 - \frac{m_\nu^2}{(Q_i - E_e)^2}}$$

(1.23)

where $p_e$ is the electron momentum, $Q_i$ is the end point energy when the daughter atom remains in the excited i-state after the decay and $\pi_i$ is the probability for the daughter atom to remain in the i-state (with
\[ \sum_i \pi_i = 1 \). \( F(Z,E_e) \) is the Fermi function which takes into account the Coulombian screening effect of the surrounding nucleus electrons on the outgoing electron. This function for the \(^{187}\text{Re}\) beta decay has been calculated by Buring [22] following the Behrens and Janecke’s tables; he gave an estimation also of \( S(E) \), the shape factor which is related to the transition type that for \(^{187}\text{Re} \quad (J = (5/2)^+) \) to \(^{187}\text{Os} \quad (J = (1/2)^-) \) is a first order unique forbidden transition (\( \Delta J = 2, \Delta \pi = 1 \)). The product \( F(E_e,Z)S(E_e) \) can be considered in first approximation constant and in the endpoint region it affects the spectrum only less than the 5\%. When the \(^{187}\text{Re} \) undergoes the beta decay, the final state is the ground level of \(^{187}\text{Os} \) atom in the rhenium crystal, therefore the energy necessary to create and position the missing electron in the osmium’s shell is to be considered. The end-point energy of the \(^{187}\text{Re} \) in the crystal is decreased respect to the decay of an isolated isotope and can be written as:

\[
E_{\text{endpoint}} = (Q - m_e c^2) - (E + E_{\text{Fermi}}) - B_{\text{lattice}} \quad (1.24)
\]

where \( Q \) is the energy available for the nuclear decay, \( m_e c^2 \) is the energy for creating the electron mass, \( (E + E_{\text{Fermi}}) \) is the electron binding energy in the crystal as sum of the electronic work function (5.1 eV) and of the Fermi energy (11.2 eV), \( B_{\text{lattice}} \) is the change in the potential energy of the isotope in the crystal due to the change of one unit of the nuclear charge. The last contribution is negligible in this case since it corresponds to about 2.6 of the total binding energy of rhenium atoms in the crystal, which have been evaluated to be 16.9 eV/atom. Then, the major contribution to the change of the end-point energy, when the decay occurs in the crystal, results to be the final state of the electron in the osmium’s shell, which binding energy is 16.3 eV. The end-point value of the \(^{187}\text{Re} \) beta decay in metallic rhenium crystal is 2487 eV [20]. Respect to the problem of the final excited states it is a worthwhile that the microcalorimetric detector with rhenium based absorber is able to measure them if their relaxation time is shorter than the heat pulse formation time, which is generated by the nuclear decay. The decay may leave the daughter atom in excited levels with half life much longer than the thermal pulse formation time, which have been measured to be 120 \( \mu s \) (10-90 %). In this case the relaxation energy is lost, because it can not be summed in the heat pulse formation. Despite the probability of half-life longer than few hundred of microseconds is small and the probability of final excited states is also negligible. Indeed, because of a very similar atomic wavefunctions and levels of Re and Os, the probability of a transitions toward un excited level is very small, being the Os excited eigenstate orthogonal to the Re ground state. An evaluation of this
probability gives us values lower than $7 \times 10^{-5}$ [21].
The possibility of a loss of energy in a dislocation of the daughter isotope
during the recoil can be excluded. The energy of the recoil is lower than
8 meV and therefore not sufficient to give rise to this process, but only
to generate phonons belonging to the elastic branches, which contributes
to the heat formation.
Another process, that in principle could smear the position of the end-
point, is the recoil-free beta decay. This effect has been not observed
until now, but it can be foreseen on the basis of the extension of the
theory of the well-known Mossbauer effect to the beta decay. In the case
of recoil-free decay the neutrino-electron couple energy is increased by
an amount equal to the nucleus recoil. Because this is a so small amount
of energy, the effect on the shape of the end-point region is negligible.
Collective excitations in the crystal can also contribute to the generation
of long life metastable states, which can trap a variable amount of energy
for a long time. Because the rhenium crystal is superconducting at tem-
perature below 1.69 K, we saw that long living quasiparticle states are
predicted from the model that extent the BCS theory to superconducting
systems out of the thermal equilibrium.

1.4.2 MANU
The measurement of $^{187}$Re beta spectrum with high enough sensitivity
to set a limit on electron neutrino mass is not trivial, calorimetric mea-
surements can be an useful method to reach a suitable energy resolution.
The choise of the absorber for the detector plays an important role.
As already cited the group of Genoa decided to work with metallic super-
conducting rhenium, motivated by the high efficiency of thermalization
($\sim 100\%$), the very low band-gap value of possible metastable excita-
tions, i.e. long-life quasiparticles.
The first pilot experiment, MANU, has been executed in 1999 as described
below, with 1 detector under measurement for about 4 months. The
group of Milano proposed to develop detector with a dielectric compound
of Re, Re$\text{AgO}_4$. The experiment, MIBETA, has been executed in the
2002 with 10 detectors with one year of data taking.
Natural metallic rhenium contains almost 63 % of the isotope 187, the
expected activity from 1 mg is almost 1 Bq:

$$A = \frac{1}{2} \frac{0.001 g}{186 g/mol} \times 6.022 \times 10^{23} \times 0.626 \times \frac{^{187}\text{Re}}{\text{Re}} \times \frac{1}{1.4 \times 10^{18}} = 0.7 \text{ Bq} \quad (1.25)$$
Z  |  75  
---|---
Isotopes | $^{185}\text{Re}$ (37.4 %), $^{187}\text{Re}$ (62.6 %)  
A  |  186.207 g/mol  
Molar volume | 11.3 cm$^3$  
El. configuration | $[\text{Xe}]^4f^{14}5d^66s^2$  
Cryst. structure | hcp  
Density | 21.02 g/cm$^3$  
Debye temperature | 417 K  
Transition temperature | 1.69 K  
RT el. resistivity | $18.4 \times 10^{-6} \Omega \text{cm}$  
Critical field | 20.0 mT (200 G)  

Table 1.2: Important rhenium features

The main properties of metallic rhenium are summarized in table 1.2, the heat capacity of the normal state is given by:

$$ C = 40.6 T^{-2} + 0.034 T^{-3} + 2290 T + 27 T^3 \mu J/(mol K) \quad (1.26) $$

where the first two terms are a representation of the nuclear heat capacity in a applied field, the third is the normal electron contribution and the last term is the lattice contribution. The heat capacity of electrons in the superconducting state for temperature below twice the critical temperature, is written as:

$$ C_{se} = a \gamma T C \ e^{-b T_C/T} \quad (1.27) $$

with $a=8.5$, $\gamma=2.3 \times 10^{-3} J/(mol K)$ and $b=1.46$.

The calculated heat capacity for 1 g of rhenium at different temperatures is showed in table 1.3. In estimating the final absorber heat capacity it is important to remember that if the detector is cooled down inside the magnetic field of the Earth 0.5 G, therefore small parts of the absorber can become normal with an enhancement of the heat capacity. Measurements of detector heat capacity done during the test of rhenium absorber in different magnetic field are discussed in chapter 4. The use of metallic rhenium as energy absorber has been disregarded since microscopic calculations from the BCS theory predict that a large part of the released energy must be trapped in quasiparticles-quasiholes pairs which live for order of seconds at low temperature as 100 mK. The Genoa group gave the proof that it is possible to work with superconducting rhenium (and also other superconductors). The specific heat measured for different pieces of rhenium kept at 95 mk ranged between 0.1 to $1 \times 10^{-12}$
Table 1.3: Heat capacity calculated with the previous relations for 1 g of rhenium


\begin{tabular}{|c|c|c|}
\hline
T (mK) & \( C_{\text{lat}}(J/K) \) & \( C_{\text{en}}(J/K) \) \\
\hline
200 & \( 1.2 \times 10^{-9} \) & \( 2.5 \times 10^{-6} \) \\
100 & \( 1.4 \times 10^{-10} \) & \( 1.2 \times 10^{-6} \) \\
50 & \( 1.8 \times 10^{-11} \) & \( 6.2 \times 10^{-7} \) \\
20 & \( 1.2 \times 10^{-12} \) & \( 2.5 \times 10^{-7} \) \\
10 & \( 1.4 \times 10^{-13} \) & \( 1.2 \times 10^{-7} \) \\
\hline
\end{tabular}

JK\(^{-1}\) mm\(^{-3}\), these values may be due to the quality of different samples measured. In this experiment a superconducting rhenium single crystal of mass 1.572 mg was used both as absorber and beta source, with an activity of about 1.1 Bq. The sensor was a neutron transmutation doped (NTD) germanium thermistor (230 \( \mu \)m x 100 \( \mu \)m x 100 \( \mu \)m). Sensor and absorber are connected with a drop of epoxy (Epotek H301-2) and the microcalorimeter was suspended by two ultrasonic-bonded Al wires 15 \( \mu \)m in diameter, which also provide the electrical connection and the weak thermal connection to the heat sink. The heat sink is a 3He-4He dilution refrigerator working at a base temperature of 60 mK.

In five months of data taking, about \( 6 \times 10^6 \) \(^{187}\)Re beta-decay events from the energy threshold of 420 eV to the end-point energy have been acquired with an energy resolution of 40.8 eV rms. The energy calibration is provided by a removable fluorescence source of Cl, Ca, and Va K\( \alpha \) and K\( \beta \) x-rays, excited by an \(^{55}\)Fe x-ray source also used for the calibration. During the absense of the fluorescence source, for low background measurements, the calibration is provided by low rate \(^{55}\)Fe x rays.

Since this was the first high statistics measurement on \(^{187}\)Re with microcalorimeters, the reduction and interpretation of the data have been carefully tested. The detector characteristics turn out to be relatively simple and well understood and the experimental spectrum follows the expected beta distribution over the full energy range of the detector. Experimental behaviors considered in the interpretation of the data, and that have been analyzed in detail, include the detector response function, the detector energy resolution, the energy calibration and detector linearity, the effect of unidentified pileup, the possible distortion of the spectrum due to electron escape, the effect of the data reduction and, in particular, of cuts in the pulse shape analysis, and the goodness of the theoretical spectral shape.

The detector response has been evaluated using the \(^{55}\)Fe K\( \alpha \) line and it
is in good agreement with a Gaussian distribution. Any low-energy or high-energy tail, if present, contributes less than 0.1% to the total counts and is negligible with respect to the statistical uncertainties.

The spectrum obtained with the fluorescence source has been used to investigate the possible energy dependence of the resolution and the detector nonlinearity. The energy resolution does not show any measurable dependence on the energy in the whole spectrum. The energy resolution is completely determined by the ratio between the pulse amplitude and the noise in the detector. The detector noise is due to phonon noise from the thermal link, Johnson noise from the sensor, and amplifier noise. All these terms are energy independent; therefore the energy resolution is also expected to be independent on the energy. The data show a detector nonlinearity well described by a parabolic distribution of the energy versus the pulse amplitude.

The main result obtained in this experiment was [24]:
- end-point energy $E_{e.p.} = (2472 \pm 4 \text{ stat } \pm 2 \text{ syst}) \text{ eV}$
- half-life $\tau_{1/2} = (4.12 \pm 0.02 \text{ stat } \pm 0.11 \text{ syst}) \times 10^{10} \text{ y}$
- $m_{\nu}^2 = -469_{-679}^{+579} \text{ eV}^2/c^4$
- $m_{\nu} \leq 26 \text{ eV}/c^2$ with 95% C.L.

An important result connected to the analysis of the rhenium spectrum was the discovery of the Beta Environmental Fine Structures (BEFS) that reveals an aperiodic modulation of the spectrum with a maximum 1% of relative amplitude ([25] and reference there in). This effect is caused by the self interference of the outgoing electron wave and the reflected one from the surrounding atomic shells. The BEFS modulations have a negligible amplitude in the end-point region and therefore they don’t create any remarkable distortion in the region interesting for neutrino mass determination.

The evolution of the rhenium experiment MANU2 is characterized by the change of sensors, the Ge-NTDs have been substituted by the Transition Edge Sensors (TES) and by the number of channels, 300 in order to have an high count rate.

The first tests were done on Al-Ag TES, the results obtained showed in [56], the best resolution with this sensor was obtained with a tin absorber and was of 13 eV at 5.9 keV. The noise analysis didn’t show a presence of unexpected noise and the sensor stability of more then nine months was measured [56] [57].

The number of channels makes this measurement more difficult, in fact several drawbacks arise both from the fact that is a low temperature measurement (300 bias and read out cables which bring heat inside the fridge) and both from the fact that all the three hundred microcalori-
In this paragraph, the author describes the present developments on the MANU2 experiment, in particular they deal with the test on new IrAu sensor and briefly talk about the new signal acquisition electronics.

### 1.4.3 163-Ho Electron Capture decay

Another interesting measurement in which cryogenic microcalorimeter play a fundamental role, is the study of the electron capture of $^{163}\text{Ho}$. The endpoint of this decay is set at 2.8 keV [26] and the energy spectrum down to 50 eV present five important lines of whose four have been already detected (see tab. 1.4).

In 1981 De Rujula [27] proposed a new experiment for measuring electron Neutrino mass by means of $^{163}\text{Ho}$ Electron Capture Decay spectrum due to the very low end point energy. In fact as for beta decay sensitivity to neutrino mass depends on the term:

$$\frac{dN}{dE_C} \propto \sqrt{1 - \frac{m_{\nu}^2}{(Q_{EC} - E_C)^2}}$$  \hspace{1cm} (1.28)

The decay may be written as:

$$^{163}\text{Ho} + e^- \rightarrow ^{163}\text{Dy}' + \nu_e$$

$$^{163}\text{Dy}' \rightarrow ^{163}\text{Dy} + E_C$$

the nucleus of a $^{163}\text{Ho}$ captures an electron of the inner shell, thus becoming $^{163}\text{Dy}'$, where the prime indicates the excited state, and emitting an electron Neutrino; this energy $E_C$ is released via X-rays, Auger electron or Coster-Kronig transition, leaving the atom in its ground state.

<table>
<thead>
<tr>
<th>Spectral line energy (eV)</th>
<th>Electron binding energy (eV) [28]</th>
<th>Excitation energy (eV) [26]</th>
<th>Experimental energy</th>
</tr>
</thead>
<tbody>
<tr>
<td>MI</td>
<td>2047</td>
<td>2029.3</td>
<td>1875</td>
</tr>
<tr>
<td>MII</td>
<td>1842</td>
<td>1824.2</td>
<td>1683</td>
</tr>
<tr>
<td>NI</td>
<td>416</td>
<td>406.7</td>
<td>379</td>
</tr>
<tr>
<td>NII</td>
<td>332</td>
<td>322.6</td>
<td>302</td>
</tr>
<tr>
<td>OI</td>
<td>63</td>
<td></td>
<td>NN</td>
</tr>
</tbody>
</table>

Table 1.4: Expected energy level for $^{163}\text{Ho}$ EC decay

meter have to work at the same temperature (small corrections can be brought by changing the bias voltage).

The calculations and measurements were carried out using the MANU2 cryogenic microcalorimeter, which is designed to detect and measure the energy emitted by nuclear reactions down to very low energies. This device is capable of resolving individual photons and recording their energies accurately. The system is sensitive to a wide range of energy levels, from thermal to keV energies, making it suitable for a variety of applications in nuclear science and technology.
The possible implication for the Neutrino physics has ruled out because the large energy difference between the highest capture line and the Q value, which give rise to a very tiny effect for finite neutrino mass. Besides that some unresolved questions arose from the results of various experiments. First of all two different experiments [28] [26] measuring the calorimetric spectrum show that the x-ray lines are shifted towards energies lower than the expected and these values are incompatible with other obtained in different experiments.

The effective decay rate of each line and the number of lines that can be resolved are close connected to a precise determination of the $Q_{EC}$. In 1996 was carried out the first calorimetric measurement of 163-Ho EC spectrum [26], in this experiment it was possible separate the MI MII and NI NII lines. Beside the good result, the measurement was limited by an energy resolution poorer than the predicted one, the dependence of the resolution on the energy and a red-shift of about 10% of the capture lines. The cause of these drawbacks were found, first of all, in the embedding methods used for the radioactive source. It was deposited as organic salt on a thin tin foil that was bent and pressed for keeping all the radioactive emission inside. This fact caused that a fraction of X-rays emitted were absorbed in the dielectric salt containing the holmium instead of being thermalized directly in the superconductor, this was the main reason for the not very good resolution obtained.

The question whether the red-shift of the capture line is caused by the dielectric medium embedding the source, or by nuclear and/or atomic physics is still open. Therefore a new experiment on holmium is needed to clarify this point.

Systematic errors which could have compromised the previous experiment can be overcome by using a calorimetric technique in which the radioactive isotope would be homogeneously dispersed inside the absorber. In order to perform a true calorimetric measurement, I have studied and developed a technique to prepare superconducting absorber containing the holmium source homogeneously dispersed. This method is based on the thermal reduction of rare earths from a liquid solution containing holmium ions. The process which leads the radioactive atoms from the ionic form to make part of a superconductor is described in chapter 5.
Figure 1.4: Theoretic spectrum of 163-Ho EC decay [27]
Chapter 2

Microcalorimeter model

2.1 Introduction

Over the last 20 years low temperature detectors have taken place in many fields of scientific research, they represent a new frontier for investigation not only in nuclear physics, but also in astrophysics, biological physics and material science [29]. They can perform an impressive energy resolution, response to non-ionizing events and with the help of new generation cryostats, a good versatility for different kind of measurements. The simplest configuration for a microcalorimeter has an absorber capable of containing the energy of an event and converting it to a temperature variation, an attached thermometer to measure this variation and a weak thermal link to a heat sink that returns the absorber temperature to the starting value.

These detector can employ many types of thermometers, the most common are thermistors, where the resistance is a function of temperature. For this kind of sensors the main drawback, since they are dissipative elements, is the Johnson noise. Other types of thermal trasducer are inductive [30] and capacitive [31], where high Q (figure of merit) values greatly reduce both power dissipation and Johnson noise. Magnetic thermometer are now very promising [32] for the impressive energy resolution obtained and for the good understanding and modelling of their behavior. In the following sections I will describe the ideal model of a microcalorimeter and discuss the noise source, in the end I will discuss some recent results obtained with different types of microcalorimeter.
2.2 The ideal model

The theory of microcalorimeter was described by Mather [33] in 1982, in this description the detector was conceived as an ideal system, but when the use of microcalorimeters became more present, several unexpected behavior came out. In order to explain the experimental results the theory was extended to contain some non-ideal effects such as thermal decoupling between the electron system and the phonon system in the thermometer, the so-called hot electron model, the thermal decoupling between the absorber and the thermometer and an additional non-ohmic behavior of the thermometer [34].

In this section I will describe the ideal model for a microcalorimeter, the thermal and electric circuits, in order to explain the equations I used to characterize the microcalorimeter for my measurements. For more detailed descriptions of microcalorimeters models, I will suggest contributions from Galeazzi and McCammon [35] [36].

An ideal detector can be represented by a discrete absorber of heat capacity $C$ in contact with the heat sink through a thermal conductivity $G$ and a thermometer always at the temperature of the absorber. The thermometer sensitivity is specified by the parameter $\alpha$:

$$\alpha = \frac{T}{R} \frac{dR}{dT}$$  \hspace{1cm} (2.1)

where $T$ is the detector temperature and $R$ the sensor resistance. The sensor is typically a resistor whose resistance strongly depends on temperature variations around the working point.

The thermal conductivity $G$, defined as:

$$G = \frac{dP}{dT}$$  \hspace{1cm} (2.2)

where $P$ is the the power dissipated into the detector, it can be expressed as a power law of the detector temperature $T$:

$$G = G_0 T^\beta.$$  \hspace{1cm} (2.3)

With no input power than the Joule power used to read out the thermometer resistance, the equilibrium temperature $T$ of the detector can be determined by integrating Eq. 2 between the heat sink temperature and the detector temperature:

$$P(T) = \int_{T_s}^{T} G(T')dT' \rightarrow (T^{\beta+1} - T_s^{\beta+1}) = \frac{\beta + 1}{G_0} P(T)$$  \hspace{1cm} (2.4)
To calculate the equilibrium temperature it is therefore necessary to solve the system of equations represented by Eq. 4, the relation between the power P and the resistance R and how the resistance R changes with temperature T.

When an energetic event, with power W, happens inside the detector, the system is out of equilibrium and the temperature increases of ΔT above the equilibrium temperature T. The total input power (P+W) is partly stored into the heat capacity of the detector, and partly flows to the heat sink through the thermal conductivity, the equation that determines the temperature of the detector T_D is:

\[ C \frac{dT_D}{dt} + \int_{T_S}^{T_D} G(T')dT' = W + P(T_D) \] (2.5)

It is then possible to express the generic detector temperature T_D as a function of the equilibrium temperature T, as \( T_D = T + \Delta T \); with this substitution the previous equation becomes:

\[ C \frac{d(T + \Delta T)}{dt} + \int_{T_S}^{T} G(T')dT' + \int_{T}^{T+\Delta T} G(T')dT' = W + P(T+\Delta T) \] (2.6)

If the quantity ΔT is small compared to T, it is possible to expand the second integral to lowest order in ΔT/T (small signal limit):

\[ C \frac{d(T + \Delta T)}{dt} + \int_{T_S}^{T} G(T')dT' + G(T)\Delta T = W + P(T) + \Delta P \] (2.7)
with \( \Delta P = P(T + \Delta T) - P(T) \). Subtracting Eq. 4 from Eq. 8, and considering that the equilibrium temperature \( T \) does not change with time:

\[
C \frac{d(\Delta T)}{dt} + G \Delta T = W + \Delta P \quad \text{with } G = G(T) \tag{2.8}
\]

In general the bias power will change with temperature, since \( R \) changes, and its expression depends on the bias source impedance. A typical bias circuit is illustrated in fig. 2. The most commonly used bias conditions are near current bias (\( R_L >> R \)) and near voltage bias (\( R_L << R \)).

Differentiating the expression for the Joule power \( P = I^2 R = V^2 / R \) and using the bias circuit in Fig. 1 it is possible to obtain:

\[
\Delta P = \frac{P}{T} \frac{R - R_L}{R_L + R} \alpha \Delta T \tag{2.9}
\]

This term is generally referred to as the electrothermal feedback term and it often plays an important role in the response of the detector. In the small signal limit the electrothermal feedback term may be written as:

\[
\Delta P = -G_{ETF} \Delta \alpha \tag{2.10}
\]

with

\[
G_{ETF} = \frac{P}{T} \frac{R - R_L}{R_L + R} \alpha \tag{2.11}
\]

With this notation and introducing an equivalent thermal conductivity \( G_{ef} = G + G_{ETF} \), named effective thermal conductivity, the equation for the microcalorimeter can finally be written as:

\[
C \frac{d(\Delta T)}{dt} + G_{ef} \Delta T = W \tag{2.12}
\]

Using Fourier transforms it is easy to solve this equation in the frequency domain:

\[
j \omega C \Delta T(\omega) + G_{ef} \Delta T(\omega) = W(\omega) \tag{2.13}
\]

whose solution is:

\[
\Delta T(\omega) = \frac{1}{G_{ef}} \frac{1}{1 + j \omega \tau_{ef}} \quad \text{with } \tau_{ef} = \frac{C}{G_{ef}} \tag{2.14}
\]

The detector system behaves as a low-pass system with time constant \( \tau_{ef} \). Since this time constant depends on \( G_{ETF} \), it is easy to see that it is shorter if the electrothermal feedback is negative (\( G_{ETF} > 0 \)), moreover the negative electrothermal feedback helps in linearizing the large signal gain and improve microcalorimeter resolution for large signal at high
count rate. In order to work in negative electrothermal feedback it is necessary to use current bias \((R < R_L)\) detector with negative \(\alpha\) and voltage bias \((R > R_L)\) detector with positive \(\alpha\).

At this point I just described how the increase of detector temperature \(\Delta T\) depends on the input power \(W\), but what is measured is the following change in sensor resistance \(\Delta R\), which is read out as either voltage or current variation, these will depend on the sensor characteristics \((\alpha)\) and on the bias circuit configuration. The output signal may be written as:

\[
\Delta V = V \frac{\alpha}{T} \frac{R_L}{R_L + R} \Delta T 
\]

\[
\Delta I = -I \frac{\alpha}{T} \frac{R}{R_L + R} \Delta T 
\]

Other three important parameter useful for the characterization of the detectors can be defined: the transducer sensitivity and the dynamic impedance. The first is a dimensionless parameter that quantify how much the output signal is sensitive to a resistance variation; using \(X\) as either \(V\) or \(I\), the sensitivity is defined:

\[
A_{tr} = \frac{R}{X} \frac{dX}{dR} 
\]

and it plays a role in the output signal, in fact it can be written as:

\[
\frac{\Delta X}{X} = \alpha A_{tr} \frac{\Delta T}{T} 
\]

The responsivity quantify the response of a detector to an incoming power \(W\):

\[
S(\omega) = \frac{\Delta X(\omega)}{W(\omega)} 
\]

Finally the complex dynamic impedance takes into account not only the resistance \(R\) of the detector, but also the effects of the electrothermal feedback; it is defined as:

\[
Z(\omega) = \frac{dV(\omega)}{dI(\omega)} 
\]

For the simple model used in this chapter the dynamic impedance as the form:

\[
Z(\omega) = R \left[ \frac{1 + \frac{\alpha}{\epsilon T}}{1 - \frac{\alpha}{\epsilon T}} + j \omega \tau \right] 
\]

All these equations describe an ideal microcalorimeter model in which the electrons and phonons are at the same temperature and the sensor
and the absorber are considered together. A first correction to this simple model is the hot electron model that considers the decoupling between electrons and phonons due to the low working temperature of the detectors.

Therefore the detector is no more described as only one system, but in this model the system of electrons and the one of phonons are thermally connected by the thermal conductivity $G_{e-l}$. Two cases may be analyzed, the first in which the electron heat capacity $C_e$ is negligible and the other with a non zero $C_e$.

A second modification to the standard model consists on considering sensor and absorber as two different systems connected by the thermal conductance $G_a$. In fact most of the detectors, and in particular the detectors fabricated in Genoa have sensor and absorber mechanically connected by epoxy glue. In this scheme the input power $W$ interacts with the absorber system which is connected to the lattice sensor system as shown in fig. 2.2. This attention in modelling the working of e

![Diagram](image)

Figure 2.2: Simple thermal circuit in the hot electron model and with absorber decoupling

microcalorimeter is important in order to understand the noise source. In fact only with the knowledge of all the components of noise will be possible to improve the performances of these detectors.
2.3 Noise

The performances of a microcalorimeter are affected by several noise sources, their effect is quantified by the Noise Equivalent Power (NEP), that is an internal power change that produces a signal equal to the total rms noise. The NEP is calculated as the ratio between the output noise signal and the responsivity of the device:

\[ NEP^2(\omega) = \frac{(\Delta X)^2}{S^2(\omega)} \]  \hspace{1cm} (2.22)

The total noise may be described as a sum of several contributions: thermal, Johnson, 1/f, the noise due to the load resistor and the amplifier noise. Usually the sum of these components are not enough to account the measured noise, in fact in a range around 1 kHz there is a not expected “hill”, that represents the so called excess noise problem. The total noise may be written as:

\[ NEP^2 = NEP^2_J + NEP^2_{th} + NEP^2_{load} + NEP^2_{amp} + NEP^2_{ex} \]  \hspace{1cm} (2.23)

where any component is defined as: The analytical form of each contribution has been studied by several researches; an important contribution to understand the thermal noise was done by Mather [33] who set the relation:

\[ P_{th} = \sqrt{4k_B G T_I^2 \left( \frac{\int_{T_{l}}^{T_{s}} (T'k(T'))^2 dT'}{\int_{T_{l}}^{T_{s}} k(T') dT'} \right)^{1/2}} \]  \hspace{1cm} (2.24)

where \( k_B \) is the Boltzmann constant and \( k(T') \) is the function describing the temperature dependence of the thermal conductivity of the heat link material. The Johnson Noise of the sensor resistance is:

\[ e_J = \sqrt{4k_B T_e R} \]  \hspace{1cm} (2.25)

where \( T_e \) is the electron temperature. The load resistor noise can be represented by a voltage noise across the detector:

\[ e_L = \sqrt{4k_B T_S R_L R} \]  \hspace{1cm} (2.26)

The 1/f noise is described as a fluctuation in the value of the resistance:

\[ \frac{\Delta R}{R} \]  \hspace{1cm} (2.27)
In the hot electron model one needs to consider the power fluctuation between the lattice and the electron system and this gives a contribution to the noise as [37]:

\[ P_{he} = \sqrt{2k_B G_{e-l}(T_e) \frac{T_e^5 + T_l^5}{T_e^3}} \]  

(2.28)

Moreover if we consider the absorber decoupled from the sensor, we have to consider also the power fluctuations between absorber and sensor lattice:

\[ P_a = \sqrt{4k_B G_a T_l^2} \]  

(2.29)

With these definitions and knowing how these noise source take place in the electro-thermal circuit, we can write the relations for all the noise contributions:

\[ NEP_{th} = P_{th}(1 + j\omega \tau_a) \]  

(2.30)

\[ NEP_t = \sqrt{\frac{4k_B T_l T_e^{3e+1}}{P \alpha^2 T_e^{3e}} \left[(1 + j\omega \tau_{e-l})[j\omega C_a + G(1 + j\omega \tau_a)(1 + j\omega \tau_l)] + (1 + j\omega \tau_a) T_l^{3e} j\omega C_e\right]} \]  

(2.31)

\[ NEP_{1/f} = \left(\frac{\Delta R}{R}\right)_{1/f} \frac{T_e T_e^{3e}}{\alpha T_l^{3e}} \left[(1 + j\omega \tau_{e-l})[j\omega C_a + G(1 + j\omega \tau_a)(1 + j\omega \tau_l)] + (1 + j\omega \tau_a) T_l^{3e} j\omega C_e\right] \]  

(2.33)

\[ NEP_{R_L} = \frac{e_{R_L}}{S(\omega)} + \frac{2I^2 e_{R_L}}{G_{e-l}(T_l)} \left[j\omega C_a + (1 + J\omega \tau_a)(G + G_{e-l}(T_l) + j\omega C_l)\right] \]  

(2.35)

\[ NEP_{amp} = \frac{e_{amp}}{S(\omega)} \]  

(2.36)

\[ NEP_{he} = \frac{P_{he}}{G_{e-l}(T_l)} \left[j\omega C_a + G(1 + j\omega \tau_a)(1 + j\omega \tau_l)\right] \]  

(2.37)
\[ NEP_a = P_a j \omega \tau_a \] (2.38)

From the knowledge of the NEP it is possible to obtain the microcalorimeter energy resolution by weighting the signal at frequency \( \omega \) with the factor \( 1/NEP^2 \) and averaging. The rms energy resolution is then:

\[ \Delta E_{rms} = \frac{1}{\sqrt{\int_0^{\infty} \frac{2\omega}{\pi NEP^2(\omega)}}} \] (2.39)

In strong electrothermal feedback the FWHM energy resolution may be written as:

\[ \Delta E_{th} = 2.35\xi \sqrt{k_B T^2 C} \] (2.40)

where the quantity in brackets is the magnitude of the TFN at the heat sink temperature and \( \xi \) is a parameter related to \( \alpha \) and \( \Delta T/T \) while is weakly dependent on the thermal link, its value is very close to one.

The parameter \( \xi \) can be written as \( 2(6/\alpha)^{1/2} \) Irwin [54] proposed a different derivation of the limit energy resolution and its result was bound to the thermodynamic limit by a factor of about \( 2/\sqrt{\alpha} \):

\[ \Delta E_{th} = \sqrt{\frac{4k_B T^2 C (1/\alpha)\sqrt{n/2}}{\sqrt{\alpha}}} \] (2.41)

### 2.4 Excess-noise problem

The predictions of the noise level of the electro-thermal models don’t account the total noise that has been measured in many TES (MoCu,TiAu). The full noise remains \( 2/3 \) times higher that the expected one. The noise power spectrum shows broad peak at high frequencies around 50010000 Hz and it is typically 4 times the Johnson noise in the middle of the transition and 20 times the Johnson noise and low resistance On the other hand Al-Ag TESs show no evidence of excess noise, in [56] the estimation of sensor noise was in good agree with the expectations. The same results has also been found by other groups working on Al-Ag TES. Several group try to explain the presence of this unexpected noise with different models. Each of them seem to find a reason in the extended heat capacity model which break the single heat capacity detector in an absorber heat capacity \( \text{abs} \) and a TES heat capacity \( \text{TES} \). This separation is necessary when the dimensions are shorter then the characteristic length \( l \) that a heat pulse propagate in a time \( \tau = l^2 c/k \), \( l = \sqrt{k/2\pi \nu c} \), with \( k \) the thermal conductivity and \( c \) the specific heat.
Another point of view is to relate the excess noise with the intrinsic superconducting properties of the TES itself. Two main (possible) source of noise have been identified: the magnetic flux motion due to self-field and external field, and fluctuation in the number of Cooper pairs. As TES are superconducting thin film, an external magnetic flux perpendicular can penetrate, the consequence is different is the film is type I or type II superconductor. In the first case the flux penetration generates highly unstable normal metal islands which can change in size and lead to resistance fluctuation in the TES [35]. In the second case the flux line are quantized and their motion generates a voltage noise in the TES.

The fluctuations of the order parameter in a superconductor at the transition has been considered as a noise source in a superconductor by Nagaev [38] and remarked by G.Seidel in the last LTD10 [39]. The DC electrical conductivity is enhanced by the formation and dissociation of Cooper pairs as was pointed out by Aslamazov and Larkin [40]. This model was improved by Maki and Thompson which considered the modification of the normal electron conductivity by the fluctuation of Cooper pairs. The spectral density for the current fluctuation on the Maki-Thompson model for \( \hbar \omega \ll k(T - T_C) \) and \( (T - T_C) \ll T_C \) is:

\[
S_{pf} = \frac{\pi^2}{128} \frac{D e^4 E^2}{\hbar} \frac{\tau_{ph} T_C^2}{k(T - T_C)} \frac{\arctan(\omega\tau_{ph}/2)}{\omega \tau_{ph}/2}
\]  

(2.42)

where D is the diffusion coefficient, \( \tau_{ph} \) is the phase coherence time of electrons and E the electric field. Comparing this with the experience on TES noise there is a good agreement with the enhance of noise lowering the temperature, but there is no evidence of the \( E_2 \) dependence. At the moment there are no direct evidence on how the excess noise is dependent on the Cooper pairs fluctuations.

The SRON group has been able to model all the main noise source in their detectors explaining the broad peak at high frequencies. Their devices consist of a Cu/Bi absorber on top of a Ti/Au TES, they are different from other group TES since they have higher resistance and therefore lower thermal conductivity that leads to the presence of different parts of the film with different temperature and weakly coupled with each other. Bergmann Tiest et al. [41] found the source of excess noise in the thermal fluctuations present in the TES itself and named the noise as ITFN (internal thermal fluctuation noise). This effect is enhanced by the presence of the absorber evaporated on the center of the TES since the heat fluctuation across the barrier between the absorber and the sensor are bigger then the one inside the sensor itself. This group found the way to reduce the noise changing the geometry of the absorber and from
a square to an absorber divided in stripes perpendicular to the current flux, obtaining a zebra-like detector.

This zebra configuration, normal metal pattern deposited on top of the TES, seems to be useful to reduce excess noise also in other different systems. Staguhn and the group of Goddard [42] use successfully the zebra configuration. Their explanation comes from the fact that placing normal metal regions in the active region of the superconducting detector, the boundary conditions for the superconducting order parameter can be constrained. Several test were done on different geometries, metal stripes were deposited parallel to the current, perpendicular and on the edges, the best results in term of noise reduction were obtained with the perpendicular configuration.

Analogous result were obtained by Lindeman and coworkers [43] with their Mo/Au TES depositing gold bars on top. Even in their tests the configuration that lead to best performances in noise reduction was the one with bars perpendicular to the electric current. They explained the results with the proximity effect beneath the gold bars forcing the supercurrent to flow in a meander path of the bilayer between the bars.

The zebra TES seems to ensure a good reduction of excess noise, even in systems that different among them; it was created to lower the ITFN of Ti/Cu TES with over imposed the Cu/Bi absorber and in that case the normal bars represent the absorber itself, in the other case the normal bar have only the effect of noise reduction. The fundamental mechanism for which the excess noise is reduced is not still known, but the effect lead by these bars is very promising.

Other method to lower the excess noise have been investigated, I still want to cite the Corbino Disk [44] and the use of small external magnetic field.

The geometry used for the Corbino disk, a disk with an electrical connection in the center and the other along the external perimeter, was devoted to eliminate the edges, avoiding all the noise causes from edge defect and magnetic flux penetration. The main drawback was the change in current density along the current direction with a consequent higher density toward the center that leads the film to the normal state in that region.

The use of small magnetic field perpendicular to the TES film was experimented at NIST [45], and they found out that a field of 130 and 210 mG significantly reduces the excess noise.

In all these method, the normal metal bars and the use of magnetic field, the $\alpha$ parameter is reduced, for example Benford [46] reports a reduction from $\alpha = 6787$ to $\alpha = 89$ and the same parameter measured by Ullom was decreased from 182 with no field to 34 with 210 mG. This states that
the method of decreasing the excess noise leads also to a reduction in detector responsivity due to low $\alpha$ values, but there comes also a question: is the reason of excess noise related to the parameter $\alpha$?

2.5 Another way of looking at excess noise problem

The excess noise seems to be strongly related to the $\alpha$ value as all the method used to decrease this noise, work in decreasing that parameter too. How to connect $\alpha$ to what happens in a superconducting film at the transition is not very easy.

Before of all it gives the information about the steepness of the transition or how fast the resistance changes with temperature. This is related to the velocity of Cooper pair condensation and then to the variation of order parameter.

Another important aspect is the use of proximity effect to lower the transition temperature of a superconducting film by means of a normal metal film in “good electrical contact”.

The TES is thermostated at the metal film transition temperature, but the temperature itself is not uniform over all the sensor due to the finite thermal conductance from the heat sink (whose temperature is fixed) to the metal film and along the film itself. For example in [?] Bruijn et al., simulated the temperature gradient along a silicon nitride membrane, the lower value is about 50 mK and the upper value 100 mK. Since the sensor and the electrical links present several interface between different metal of the type N-N ans N-S (N=normal metal, S=superconductor), an induced current may appear due to the temperature difference between two point.

In these years studies on mesoscopic structures pointed out the interesting behavior of the thermopower in N-S structures. In particular interesting measurements come out from Andreev interferometers experiments [48].

We talk about thermopower when an electric field arises in a non uniformly heated conductor, which is proportional to the temperature gradient and can be written as:

$$E = QT$$

where Q is known as the thermopower. In normal metal Q is determined by a derivative of the logarithm of conductivity $\sigma$ with respect to the
energy $\epsilon$ taken at the Fermi level:

$$ Q = \frac{\pi^2 k_B^2 T}{3 e} \left( \frac{\delta \ln \sigma}{\delta \epsilon} \right)_{\epsilon = \epsilon_F} $$

(2.44)

The thermopower of a normal metal in contact with a superconductor will be strongly modified by the superconducting proximity effect and it can be orders of magnitude larger than predicted by the previous equation. In the work of Parsons et al. [49], it is shown how the thermopower is sensitive to the derivative of the resistance in function of the temperature, For low applied voltage the energy window available for quasiparticles is set by the temperature and therefore $(\delta \sigma / \delta \epsilon)_{\epsilon = \epsilon_F}$ can be substituted by $(1/R)(dR/dT)$ obtaining:

$$ Q = \frac{\pi^2 k_B^2 T}{3 e} \frac{1}{R} \frac{dR}{dT} \rightarrow Q = \frac{\pi^2 k_B^2}{3 e} \alpha $$

(2.45)

Therefore there will be an internal electric field proportional to the thermopower to add to the external polarization voltage. But it is necessary to have a temperature gradient inside the TES itself to apply this model. It is important to remember that a microcalorimeter has a weak thermal link to the heat bath, and this is the reason for a temperature difference between the lead wire and the main sensor body. In fact there is a very small power dissipated in the TES, the well known Joule power, due to the low residual resistivity, at very low temperature as are the microcalorimeters working temperature, this will provide a temperature difference that comes from the equation:

$$ \Delta P = G \Delta T $$

(2.46)

where $G$ may be roughly calculated from the residual resistivity using the Wiedemann-Franz law. Knowing the $\Delta T$ it is possible to estimate the current flowing in the TES due to the thermopower:

$$ \Delta I = \frac{E}{R} = \frac{Q \Delta T}{R} = \frac{C \alpha \Delta T}{R} $$

(2.47)

with $C = \frac{\pi^2 k_B^2}{3 e}$.

This is a simplified analysis which doesn’t consider any other thermal conductance that the one of the TES metal film while one would have to consider also the conductance of the electric link, the conductance between the film and the substrate and the on of the substrate itself. Anyway just in the simple case it is possible to make some estimation. In [47] the temperature gradient in the sensor zone on a silicon nitride
membrane can reach a range of 20 mK. In the work of P. Virtanen and T.T. Heikkila the thermopower in N-S structures can reach values of order of 10 \( \mu \text{V} / \text{K} \). If we consider a \( \Delta T \) of 1 mk and the previous Q value it comes out voltage difference of 0.01 \( \mu \text{V} \) that is not negligible.

This is a first and rough analysis on how the thermopower may influence the noise in TES sensors. We are developing more accurate models to include all possible temperature variation causes on the sensor in order to quantify this effect.
Chapter 3

Sensor

3.1 Introduction

The thermal sensor rules the passage from the energy released in the detector to the electric signal. The most common sensors are thermistors, which have maximum sensitivity in the 0.1 K temperature range. The most successful types are the: Si implantation doped, the Ge Neutron Transmutation doped, the Tansition Edge Sensors, the Metallic Magnetic calorimeter, the Kinetic Inductance Sensors. In this thesis I will deal mainly with TES sensor but I will describe also the features of magnetic sensors.

In this chapter I will describe the developments of TES for the project MANU-2 of INFN. AlAg and Ir-Au based TES have been investigated. Al-Ag are interesting for the very high thermal responsivity, the easy way of production, the absence of excess noise. In particular I will deal with the models of this system, especially for what concern the phenomena at the interface between two layers.

3.2 Transition Edge Sensors

Among various sensors for low temperature detector, TES are without any doubts the most studied [29]. Currently, there is great interest in superconducting sensors for spectroscopy detectors, both for particles and single photons [51] [53] [52], due to their lower noise, fast response and excellent energy resolution. TES are made of metallic thin film which undergoes the transition to normal to superconducting state at temperatures needed for a proper detector operation. The transition temperature is tuned by using the proximity effect between a normal and a
superconducting metal. The width of the transition from normal to superconducting state sets in few mK or lower, providing a strong resistance versus temperature dependence giving rise to a very high thermometric responsivity $\alpha = T/R \, dR/dT$. The typical read-out scheme needs for a low impedance and low noise trans-resistance amplifier. DC - Superconducting QUantum Interference Device (DC-SQUID), arranged as current amplifier, allows to perform the best noise matching with the sensor. The performance of TES, in term of energy resolution and count rate capability, takes advantage of the strong electrothermal-feedback (ETF) achievable by the present techniques [54].

The TES is held in equilibrium on its superconducting transition by applying a DC-voltage bias across the metal film and linking it to a cold bath, so that, the TES Joule heating exactly balances the heat flow into the bath. When an impulsive stimulus is applied, the temperature of the TES, and thus its resistance, increases causing the generation of a current signal, in form of impulsive reduction of current flowing in the TES. As high is the negative ETF, as faster is the restoring of the stationary working temperature. In fact, after the energetic event, the increase of resistance leads to a Joule power $V_b^2/R$ decrease, lowering the TES temperature faster than the naturally time constant determined by the heat conductance to the heat sink. The negative ETF allows the formation of a stable stationary working point and contributes to the linearization of the response.

The proximity effect permits to produce superconducting films with suitable transition temperature due to the effect that a normal metal has on a superconductor when they are in good electric contact. In the laboratory practice this is obtained by growing over a superconducting film, a layer of a normal metal. The main features of proximity effect and the models developed to calculate the critical temperature of the film are described in Appendix A.

Several systems are used for TES preparation, the most common are Mo-Cu, Ti-Au, Ir-Au and in the way to be abandoned, Al-Ag due to several problems found [55]. Recent measurements done by the group of Genoa, pointed out that detector with Al-Ag TES were stable for long period and performing good results [56]. The behavior of these sensors in not fully understood [35], above all, concerning the presence of noise not predictable from theoretical calculations. This problem will be discussed in the next chapter regarding the excess noise.
3.3 **Al-Ag TES development**

In these last years, the use of AgAl bilayers (multilayers) has been abandoned by several groups working on low temperature detectors for several reasons [55] such as interlayer diffusion and chemical activity, which prevent the use of standard microlitographic processes. We have extensively studied Ag-Al pursuing the aim to solve the problem of instability by inter-diffusion and chemical activity. The work has been motivated also by positive results obtained with Al-Ag TES which showed very long term stability: undetectable changes of transition temperature and transition width up to nine months. Remarkable performances have been reached with these TESs, in fig. 3.1 is shown the best spectrum obtained with $^{55}Fe$ source [56]. The resolution reached was 13 eV FWHM at 5.9 keV, the working temperature was 152 mK and the absorber had 100% stopping power.

Moreover the same detector was used over a period of nine months suffering several cooling down and it was also moved to Grenoble to ESRF for x-ray material analysis [57]. This was a good demonstration of stability of Ag-Al TES over a long period of time and therefore a good sensor for experiment in which the acquisition lasts several months as MANU2. In order to have the reproducibility and to obtain good performances, several tests have been done to understand the physic that rules this system.

For MANU2 experiment the working temperature must be not much below 100 mK, in fact it has been shown in [58] that below that temperature the detector response gets worse, due to bad thermalization of the energy released in Rhenium absorber. The first tests were dedicated to verify how the film transition temperature depends on the thickness of the layers. Consequently the reasons causing the worsening of the sensors were investigated, in particular I have analyzed the damages produced by the fabrication processes such as photolitographic processes for patterning the silicon wafer and the evaporated films, the conservation of these samples after preparation and the diffusion process at the interface of the layers. The method used for this analysis was X-ray Photoemission Spectroscopy (XPS).

### 3.3.1 Al-Ag TES: superconducting transitions

The superconductive transition temperature of Ag-Al in the proximity regime can be predicted by several models, that are dependent on the films’ parameters. In order to apply one of these models, it is important
Figure 3.1: $^{55}\text{Fe}$ energy spectrum obtained with $\mu\text{c46}$. The energy resolution is 13 eV at 5.9 keV. [56]

to know the values of the Debye temperature $\Theta_D$ and the coherence length $\xi$ for Al and Ag (see table 3.1). Five samples were prepared in which the thickness of the superconducting layer was varied. The TESs produced in the laboratories of Genoa consist of four layers forming the sequence Ag-Al-Ag-Al: the first two are thicker, then determining the transition temperature, while the last two act as protective layers. The
first protective layer, made of silver serves as protection of the main aluminum film against fast and deep oxidation, which are activated by the wet PMMA stripping processes. The second protective layer is a sacrificial aluminum film, which acts as barrier between the PMMA and the silver layer. In table 3.2 the thickness and the measured transition temperature are presented (the measurement of $T_C$ was done few days after the evaporation).

The film thickness used for Ag-Al film allows us to use the Cooper limit to estimate the law ruling the lowering of transition temperature by changing the proportion between the normal layer thickness $d_N$ and the superconducting layer thickness $d_S$. In fact the Cooper limit is used when the film thickness is smaller then the coherence length of Cooper pairs in the normal metal and in the superconductor in a way to consider the order parameter $\Delta$ constant in the whole thickness. Therefore the spatial pair interaction is defined as:

<table>
<thead>
<tr>
<th>Element</th>
<th>$\Theta_D(K)$</th>
<th>$\xi(A)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag</td>
<td>225</td>
<td>3000</td>
</tr>
<tr>
<td>Al</td>
<td>428</td>
<td>16000</td>
</tr>
</tbody>
</table>

Table 3.1: Debye temperature and coherence length for silver and aluminum
Table 3.2: Ag and Al film thicknesses and corresponding transition temperature of the proximity film

<table>
<thead>
<tr>
<th>Ag1(Å)</th>
<th>Al1(Å)</th>
<th>Ag2(Å)</th>
<th>Al2(Å)</th>
<th>$T_C$ (mK)</th>
</tr>
</thead>
<tbody>
<tr>
<td>800</td>
<td>300</td>
<td>50</td>
<td>50</td>
<td>55</td>
</tr>
<tr>
<td>800</td>
<td>315</td>
<td>50</td>
<td>50</td>
<td>90</td>
</tr>
<tr>
<td>800</td>
<td>350</td>
<td>50</td>
<td>50</td>
<td>148</td>
</tr>
<tr>
<td>800</td>
<td>380</td>
<td>50</td>
<td>50</td>
<td>210</td>
</tr>
<tr>
<td>800</td>
<td>400</td>
<td>50</td>
<td>50</td>
<td>250</td>
</tr>
</tbody>
</table>

$$[N(0)V]_{eff} = \frac{d_S [N(0)V]_S}{d_S + d_N}$$ (3.1)

This potential replaces the BCS potential in the relation to have the transition temperature:

$$T_C = 1.14 \Theta_D e^{-\frac{1}{[N(0)V]_{eff}}}$$ (3.2)

This law fits the data of the transition temperature vs the thickness of aluminum in the Cooper limit with free parameters $\Theta_D$ and $[N(0)V]$. In fig. 3.3 are shown the experimental data and the fit. The good agreement of this relation with the data gave us the way to calculate the relative film thickness for a wanted transition temperature of the sensor. The equation of the curve as function of the aluminum thickness is:

$$T_C = 1.14 \times 200 \times e^{-\frac{1}{0.425}} e^{-\frac{850e}{0.425}}$$ (3.3)

### 3.3.2 XPS analysis of Ag-Al TES

In order to understand and improve the performances of our sensors, several tests were carried out. I have undertaken my investigation when the Al-Ag TESs were already used in Genoa and the 4-layer structure have been already proposed to solve the production problems [56] (in this work and in [57] there are the procedures and products used for film preparation). I have executed the film diagnostic and the film analysis in order to investigate the causes of degradation. In [55] Holland et al. pointed out that the use of Ag-Al system for TES sensor was not reliable. They found out chemical degradation due to the preparation procedures and, above all an impressive interlayer diffusion. This latter is the reason for degradation during time of the superconducting transition which leads to a modification of the working point over long periods therefore it
represents a drawback for using this sensor for long time measurement. In this work I carried on X-ray Photoemission Spectroscopy on TES, produced with our devices, taken from silicon wafers as the tested ones. With this technique it is possible to analyze the chemical composition (even the presence of bonding states due to compounds formation over the surface) looking at the energy of electrons excited by x-rays [59].

First of all my attention was addressed to look for chemical damages. I classified them into two groups: damages due to preparation procedure and damages due to casting. In order to understand the causes of the worsening, some of the sensors were prepared for the XPS analysis. The measurements were carried on in collaboration of Dr. Andrea Chinarini of the INFN of Genoa.

A first set of measurements were addressed to see which elements are present on the surface. In fig. 3.4 one of these measurements is presented, in each box the relative abundance of one element on the TES surface is shown. In this group, two elements appear as impurity on the film: sulphur and boron. The first comes from the photolithographic process, since it is present in the triphenylsulfonium hexafluoroantimonate onium salt, one of main component of photoresist [60], while the second comes from the evaporation since the silver is evaporated from BN liner.
Both elements are bound to silver, but while sulphur is more superficial, boron is present over the whole layer. This means that if the damage by the presence of S may just change the relation between thicknesses and transition temperature, boron may lead the film to be not superconducting depending on its concentration.

For the casting contamination, the aluminum passivation is the more relevant effect; this is a superficial effect not involving the internal part, it behaves as a protection layer just decreasing the effective superconducting thickness. Other smaller effects are the carbon contamination and slight acid etching. After these measurements it was possible to improve the fabrication of TES sensors especially for the evaporation of silver.

Another investigation I carried on concerns the diffusion process between aluminum and silver layers. The analysis was done with sputtering cycles over the sensor surface followed by XPS acquisition (we took into account the fact that for very precise measurement the results obtained in way may be a little misrepresented since the sputtering may enhance the diffusion and because the Ar ions scatter in different way with Ag and Al). The result obtained were without any doubt (for the evidence of the

Figure 3.4: XPS surface chemical analysis. Each picture shows the presence of one element; the carbon and oxygen come from atmosphere contamination, the sulfur in the same zones where silver is present comes from the photoresist

50
effect) the sign of a huge diffusion process. In fig. 3.5 the XPS analysis
done over the whole thickness of a sensor is shown. The sample used in
this measurement was taken from the same wafer of a well working TES;
the thickness of the layers were (from the outer to the inner): 50 Å Al +
50 Å Ag + 350 Å Al + 800 Å Ag.

Another diffusion process is also present at silver-silicon interphase,
but this works only in reducing the effective normal layer properties.
The diffusion is a natural process and can not be kept away without
changing the system, this lead several research groups to abandon Ag-Al
system because the transition temperature was not stable during time.
The performances we observed with this system denote a completely dif-
ferent behavior of various Ag-Al TES, this probably is due to the amount
of aluminum and silver used and to their proportions. A model explain-
ing how make Al-Ag a stable system is shown in the next paragraph,
beginning from the feature of Al and Ag.

3.3.3 Al-Ag system

Both aluminum and silver have fcc crystallographic structure, their lat-
tice parameters are respectively 2.889 Å and 2.863 Å, so the lattice mis-
match is only 0.9%. The aluminum surface free energy is 1.16 J/m² and
it is smaller than the silver one, 1.25 J/m², for this reason Al has the
tendency to segregate to the surface of Al-Ag alloys (≥ 3 % of Ag).
Since the mixing heat between the two metals is negative (-0.61 eV),
Al and Ag can easily form alloys of any proportions in bulk phase. In
fig. 3.6 the phases diagram of Ag-Al system is shown, the solubility of
aluminum in silver is high even at room temperature, while at the same
temperature the solubility of silver in aluminum is negligible. The Ag-
Al equilibrium ground states are therefore fcc-based Ag, complex cubic
βMn-based Ag₃Al, hcp Ag₂Al and fcc-based Al. The Al-rich Ag-Al alloy
segregates into the fcc Al-rich terminal solid solution and the hcp inter-
mediate γ phase. No phases other than hcp and fcc are known in the
composition between Ag₂Al and Al.

For TES preparation it is very important to understand what happens
at the interface between Al and Ag. In fact it is of basic importance
since the sharpness of interfaces is one of the main parameters for a good
working of proximity effect, while diffusion processes may drive to bad
final results. Several works were done in order to understand what hap-
pens during the deposition of Ag on Al and viceversa, I describe briefly
the most relevant ones there after.
Looking at the phase diagram, I want to stress the presence of δ phase
Figure 3.5: Changes in concentration with depth of aluminum, silver, oxygen and silicon for Al-Ag TES
since it has a wide range of solid solution, it is stable at room temperature and therefore it seems to be reasonable its formation at the interface during the deposition of Ag on Al and Al on Ag.

In fact a very interesting result was obtained by Kim et al. [62], they studied the initial growing modes of Ag on Al(111) and their main success was to detect the formation of $\delta$ phase during the evaporation of the first layers. In this work a sample of Al(111), set in a vacuum chamber at 4-7 $10^{-11}$ torr, was prepared with several cleaning cycles alternating Argon sputtering and annealing at 750 K until no contaminants were detected over the surface. They made both structural analysis, by means of Low Energy Electron Diffraction (LEED), and chemical analysis, by means of XPS, during evaporation of Ag onto Al(111) bulk. The measurements were performed both at room temperature and at 50 K in order to understand the role of temperature in the process kinetic.

After the cleaning of the aluminum substrate, the LEED picture shown the three-fold symmetry typical of fcc(111) as Al is. The silver was thermally evaporated at a rate of 1-1.5 ML/min (Mono Layer per minute).
Just from the beginning of the evaporation, the LEED structure became weak until disappeared between 2 and 4 Ag ML. After the fourth layer the structure is well defined again, but this time it has six-fold symmetry. There are two possible reasons for this: or Ag film is composed by two twin-domains with fcc stackings rotated of 180°, or the intermetallic alloy δ phase is formed.

This fact was explained by the XPS chemical analysis. Looking for the energy shift of the binding energy lines as a sign of alloy formation, they saw that the Ag 3d_{5/2} line, sited at 368.2 eV for metal Ag, was shifted towards higher values, of about 0.5 eV. This behaviour is the same expected for Ag in Ag_2Al.

As the deposition proceeds, the value of the energy peak comes back to the one corresponding to metal Ag. This observation suggest that the alloy formation is present just at the interface.

An similar result was obtained by Wytenburg [63], in his experiment Al was evaporated onto Ag(111) and onto Ag(110). Analyzing the surface during evaporation with Auger Electron Spectroscopy (AES) a peak at energy of 61 eV appears and it was attributed to I_{2,3}N_{4,5}N_{4,5} Auger transition of aluminum in Ag_2Al.

The explanation proposed by Kim has not been completely accepted by Fourneé et al. [64] which were not convinced about the δ phase formation. Analyzing the surface with an STM they saw that the loss of coherence in Kim’s experiment would be due to island formation and the energy shift for Ag d_{5/2} was probably explained by Shockley partial dislocations. Anyway their measurements didn’t totally exclude the formation of the δ phase.

Another confirmation of the δ phase formation due to Al migration through Ag is given by Aswal et al. [65]. They investigate the Al migration in Ag evaporating Ag(100 nm)/Al(10 nm) on Si(111) substrate by means of molecular beam epitaxy. These bi-layers were then annealed in situ at different temperature between 25° C and 500° C under UHV (10^{-8} Torr). XPS analysis clearly reveals Al migrates to the film top where reacts with oxygen, even at room temperature. In this work as it was previously found by Kim, the Ag lines have a slight shift in binding energy to higher values and the explanation proposed was the appearance of the Ag-Al alloy, since, looking at the phases diagram this inter-metallic is stable at room temperature. Another important aspect that they measured is the room temperature resistivity; the theory suggests that the Ag resistivity will increase when it alloys with Al. The measured resistivities are 1.76, 3.90 and 4.57 μΩ cm respectively for an annealing temperature of 25° C, 250° C and 500° C, which denote a better alloy formation with
higher temperature.

3.4 Preliminary considerations on Al-Ag TES

From the results obtained with the XPS analysis and from the previous cited works in which Al-Ag TESs were investigated, it seems not straightforward to obtain good performances with Al-Ag TES, in fact they are easy chemically contaminated and the diffusion processes are naturally present.

The estimation of the transition temperature was made in the Cooper limit in which is considered the average pair potential, depending on the amount of metals and not on interface sharpness, therefore it can also represent the case in which the pair potential of Cooper pair in aluminum is modified by the alloy with silver. All the results indicate the our Al-Ag TESs are working in presence of a huge diffusion between the layers, situation considered not ideal for good working sensors. The question is what made our sensor working over nine months with identical performances when other groups found a catastrophic degradation.

A possible way to explain these results, is to consider the amount of Ag and Al we used. In fact it is without any doubt that the diffusion process can not be stopped easily, and we didn’t anything in that sense. The XPS measurements show a total mixing of Al and Ag. Looking at the phase diagram the only inter-metallic stable phase is the hcp $\delta$ and from the superconducting material handbook [66] it appears that this phase is superconducting too. In fact it is reported that $Ag_{0.70...0.60}Al_{0.30...0.40}$ has a transition temperature depending on the concentration of Ag for example it was 0.131 K for $Ag_{0.667}Al_{0.333}$ and 0.111 for $Al_{0.625}Al_{0.375}$ [67]. So there is a wide range of concentration in which the Al-Ag inter-metallic is superconductor.

To have an idea of what happens, let’s thing that all the Al get mixed to the Ag, so if the proportion falls into the right range we will have a stable TES. The atomic proportion of Al and Ag can be easily evaluated by the thicknesses of the layers, in fact the lattice parameter of both aluminum and silver is nearly the same, therefore the number of atoms will be proportional to the thickness. Considering the usual thicknesses of Al-Ag TES we have: $800 + 50 = 850$ total thickness of Ag $350 + 50 = 400$ total thickness of Al $850 : 1250 = 0.68$ proportion

This value falls inside the $\delta$ phase range. In particular the measured tran-
sition temperature of 152 K is in very good agreement with the values obtained in [67] (see fig. 3.7).

Figure 3.7: The point at 68 is the temperature transition of the TES while the other point are δ phase bulk temperature transition

3.5 Next generation Ag-Al TES

It seems that we have experienced the case in which the stability of Ag-Al samples is due to the formation of the δ phase. This gave me the suggestion to fabricate a TES in which the superconducting film was the intermetallic itself evaporated directly from a bulk alloy. In this way the system should be stable against diffusion processes.

Another important advantage to substitute the alloy TES to the proximity effect TES is the uniformity of behavior, in fact MANU2 project needs of 300 sensors working at the same temperature with nearly the same performance, this means that the working point has to be the same for all the 300 detectors. The superconducting transition of sensors taken from the same wafer is not always reproducible, since small defects of the edges or over the film thickness may change the shape of the transition or the critical temperature itself.

The film growth of a metallic alloy will provide a more constant behavior over the whole wafer, since the transition temperature depend on the alloy itself, this offers the possibility to have from one evaporation about one hundred sensors hopefully with the same final parameters.
3.5.1 $\delta$ phase characterization

To test this hypothesis, the first step was the preparation of the bulk alloy, that was executed in collaboration with Dr. Pietro Manfrinetti of the Department of Chemistry (University of Genoa). The composition chosen was Ag-35 at. Al, almost in the middle of the $\delta$ phase composition.

A tantalum crucible was filled with 2.814 g of silver and 0.379 g of aluminum, this was closed by arc welding under the flow of pure argon. At the beginning it was heated at 1200 °C in an induction furnace, then using a resistance furnace, its temperature was increased in several steps to 620 °C and kept for three days. At the end of this period the temperature was decreased again in several steps. The bulk was characterized by X-ray diffraction technique and the pattern showed in fig. 3.8 show the presence of only the hcp phase, with no other component.

![X-rays pattern of Ag-35 at. Al powders](image)

The lattice parameters calculated from this measurement were: $a=2.876 \text{ Å}, c=4.608 \text{ Å}$ in good agreement with the expected one [68]. From this bulk I cut a small rod for the superconducting transition measurement. In fig. 3.9 the transition is reported and the critical temperature measured is about 120 mK.
Table 3.3: Miller Index and relative intensity for Ag$_2$Al x-ray pattern

<table>
<thead>
<tr>
<th>h k l</th>
<th>$2\theta$</th>
<th>Intensity</th>
</tr>
</thead>
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<tr>
<td>1 0 0</td>
<td>36.03</td>
<td>244.6</td>
</tr>
<tr>
<td>0 0 2</td>
<td>39.17</td>
<td>257.3</td>
</tr>
<tr>
<td>1 0 1</td>
<td>41.19</td>
<td>1000</td>
</tr>
<tr>
<td>1 0 2</td>
<td>54.27</td>
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<td>1 1 0</td>
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<td>25.2</td>
</tr>
<tr>
<td>1 1 2</td>
<td>78.39</td>
<td>188.4</td>
</tr>
<tr>
<td>2 0 1</td>
<td>79.72</td>
<td>135.2</td>
</tr>
</tbody>
</table>

3.5.2 TES film deposition

In order to evaporate an alloy keeping constant the stoichiometric proportions a possible way is the Pulsed Laser Deposition (PLD). Because
<table>
<thead>
<tr>
<th>Parameter</th>
<th>Sample I</th>
<th>Sample II</th>
<th>Sample III</th>
<th>Sample IV</th>
</tr>
</thead>
<tbody>
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<td>$8 \times 10^{-8}$</td>
<td>$4 \times 10^{-8}$</td>
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<td>$5 \times 10^{-7}$</td>
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</tr>
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<td>RT</td>
<td>RT</td>
</tr>
<tr>
<td>heater</td>
<td>RT</td>
<td>RT</td>
<td>RT</td>
<td>RT</td>
</tr>
<tr>
<td>theor. thickness (nm)</td>
<td>20</td>
<td>50</td>
<td>100</td>
<td>450</td>
</tr>
</tbody>
</table>

Table 3.4: Parameter for hcp $\delta$ phase Pulsed Laser Deposition. The thickness was estimated from a previous calibration of 22 nm in 10 minutes. RT= Room Temperature

our PLD system was not working at that time we used the facility belongs to LAMIA laboratories of the INFM (National Institute for Matter Physics) of Genoa. The target was prepared following the same procedure described in the previous section and then pressed to have the suitable diameter. The substrate was a silicon wafer with 110 orientation, structured with UV lithographic technique in order to have the meanders shape sensors [56], with 1 mm length and 20 $\mu$m width. Four different kind of samples were evaporated in which the thickness of the films was varied. This was done to test how the features of the superconducting transition are affected by the film thickness. In each evaporation several samples were prepared in order to make different tests. In table 4.3 the evaporation parameters are reported, the substrate was not heated during the evaporation.

The first test was done evaporating at room temperature looking for $\delta$ phase formation keeping low the Al oxydation. At a first sight the film was not perfect, but several ripples were present. The reason was the degradation of the negative photoresist during the evaporation, in fact the defects were present in the area covered with the resist film. After the acetone etching, to dissolve the resist and the metallic layer in excess, the meanders appear with no evident defects and showing a good adhesion to the substrate.

3.5.3 $\delta$ Ag-Al TES first results

The first measurement was the determination of the crystal structure in order to verify the presence of the $\delta$ phase. Actually the x-ray were not as the expected, in fact even if the Ag-Al alloy were detected, the signal
was very weak and a huge cubic phase appeared, denoting that the two elements at room temperature prefers not to alloy (see fig.3.10). Low temperature resistance measurement to test the superconductivity was tried; one of the sensors evaporated with 100 nm thickness, was prepared in a sample holder with two bonding Al:Si wires as electrical contact and set in a dilution refrigerator. The value of the resistance at room temperature was 25.6 Ω and at the LHe was 18.04 Ω, lowering the temperature to 55 mK only the bonding wires transition has been seen. The reason for this failure may be found in the evaporation procedure, at room temperature the Al and Ag, even if mixed in the right proportion prefer to cluster among atoms of the same type. From the work of Aswal [65] in which the alloy formation is enhanced by the high temperature and from the bad result obtained, a second evaporation was performed onto a heated substrate, the temperature was about 500 °C. The drawback of this technique is the possible aluminum oxydation due to the residual oxygen in the vacuum chamber. For this test no structured Si wafer was used and the evaporation was done over the whole wafer. The parameter of the evaporation were similar to the previous time and the process last 40 minutes. The x-ray
analysis showed both the presence of the cubic phase and the hexagonal, but there was also an evident signal of amorphous crystallization (see fig. 3.11 and fig. 3.12). Comparing these structures to the ones obtained without the heated substrate, the $\delta$ phase signal is more evident.

Figure 3.11: x-ray pattern corresponding to the film evaporated with the substrate kept at about 500$^\circ$C.

A piece of the film has been cut and prepared for a resistance measurement. The surface of the sample didn’t show a metallic reflectivity, at the optical microscope small shining drops appear on the surface while the background was opaque. The dimensions of the piece prepared were $0.8 \times 0.1 \times 1 \times 10^{-6}$ cm$^3$ and the expected resistance would be about $1 \ \Omega$ but the room temperature resistance was of $1.7 \ \text{K}\Omega$ probably due to the presence of oxidized parts. Of course the high value of the resistance is not very promising, but I think it is worthwhile to try a surface cleaning with ion sputtering in order to see if it is possible to remove the oxyde layer and after that look for a superconducting transition measuring the resistance at low temperature. Otherwise a possible way of reducing oxygen etching will be, in the next evaporations, to use a oxygen getter as yttrium, or evaporating it just before the aluminum alloy evaporation, or having yttrium metallic film inside the chamber itself kept at temperature high enough to enhance the reactivity but to keep the vapor pressure order of magnitude smaller than the vacuum inside.
Figure 3.12: Particular of the x-ray pattern in which the three structures corresponding the the $\delta$ phase are present, but it is still evident the presence of a peak of a cubic phase.

Taking into account that the PLD may be the not perfect method to evaporate aluminum alloy, other possibility have to be considered. The first is the contemporary evaporation from two effusion cells of aluminum and silver stabilizing the crucibles temperature in order that the silver vapor pressure is twice the aluminum vapor pressure to have a gas atmosphere with about the composition of $\text{Ag}_2\text{Al}$.

The second method is the evaporation of Al Ag multilayers (with very thin single layer) maintaining the Ag thickness twice the Al (in fact the lattice parameter of both is almost the same and therefore the atomic concentration may be controlled by the thicknesses). Actually this latter method is a natural consequence of what has been done for the Al-Ag TES prepared in the previous years, only the number of layers will be increased in order to enhance the diffusion between layers and reach the $\delta$ phase stability.

### 3.5.4 Understanding microscopic properties of Ag-Al system: Monte Carlo simulations

The possibility of describing growth mechanism of thin films and understanding the behavior of atoms during the deposition on substrate is very
attractive since it could help in the interpretation of experimental results
and in the devise design improvement.
In collaboration with Dr. Riccardo Ferrando of the Genoa University the
investigation of microscopic mechanism ruling the diffusion between the
layers has been begun. The study was performed by means of Molecular
Dynamic (MD) Simulation, a technique suitable to calculate equilibrium
and transport properties of a classic many-body system, in which the
dynamic of the bodies can be described by Newton equations. Once the
initial conditions are given, the MD program can make previsions on the
evolution of systems and can calculate the value of observables when a
new configuration is reached. The heart of a program, simulating metal
properties, is the parametrization of the interaction potential. There ex-
ist several frameworks in which pair potentials can be calculated such as
the effective medium theory, the glue model.
In order to simulate Ag-Al system two different potentials have been
taken into account, the first in the EAM formalism [69] provided by C.L.
Rohrer which has been described in [70]; the potential parameters pro-
vided by the author herself didn’t show good result in calculating the
static properties of aluminum and silver. The second, from which the
most encouraging results have been obtained, is written in the frame-
work of the second moment approximation in the tight binding model,
developed by Rosato, Guillope’ Legrand (RGL) [71]. The parameters for
aluminum and silver have been provided by Prof. Papanicolau [72] of the
University of Ioannina (Greece) [72].
The first test on Al-Ag potentials lead to abandon the EAM formalism
since the results obtained in fitting observables such as cohesion energy,
surface energy and jump barriers were not correct. Therefore all the
attention is now devoted to characterize the potential written with the
parameters calculated by Prof. Papanicolaou.

3.6 Other TES possibilities

The Ag-Al system is not the only one studied by the Genoa group as TES
superconducting films, Ir Au TES has been also prepared with good film
quality and dilute alloy of Al:Mn and Zn:Mn were tested as possible
sensors. In the next sections I will briefly describe these two possibilities.

3.6.1 IrAu TES

Ir-Au system is very attractive for long term measurements since it has
a good stability over long periods and they are very resistant against
mechanical and chemical damages. Two different methods for iridium thin film preparation were tested: the Pulsed Laser Deposition (PLD) and the electron beam evaporation. Thanks to the collaboration with the group of prof. F. von Feilitzsch, we were able to test Ir-Au TESs, that have been prepared in the laboratories of E15 Department of the Technische Universitaet Muenchen. The procedure and film characterization are reported in [57].

![Figure 3.13: Superconducting transition of IrAu TES used in uc54](image)

Several tests have been performed to characterize IrAu sensors prepared with both procedures, in this thesis I will only describe in chapter 6 the best result obtained in which a $^{55}Fe$ spectrum has been acquired and the resolution of 5.9 eV has been reached. The sensor used in that test has been evaporated by electron gun on silicon substrate, the structure of the film was the following:

$$(2 \text{ nm Ir} + 20 \text{ nm Au})_{baked} + (100 \text{ nm Ir})_{RT}$$

The first thin layer of iridium has been necessary as stick layer for gold and in order to have a good adhesion they were baked at 350°C, the main iridium layer was evaporated at room temperature. The base pressure of the vacuum chamber was $8 \times 10^{-10}$ mbar using LN2 shield. The
Photolithography

<table>
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</tr>
<tr>
<td>Spin</td>
<td>1000 rpm (30 s)</td>
</tr>
<tr>
<td>Baking</td>
<td>100°C C (300 s)</td>
</tr>
<tr>
<td>Exposure</td>
<td>UVA (20 s)</td>
</tr>
<tr>
<td>Developer</td>
<td>ma-D330 (30 s)</td>
</tr>
</tbody>
</table>

Plasma etching

<table>
<thead>
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<th>Value</th>
</tr>
</thead>
<tbody>
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</tr>
<tr>
<td><strong>Acceleration voltage</strong></td>
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<tr>
<td><strong>Current</strong></td>
<td>35 mA</td>
</tr>
<tr>
<td><strong>Time</strong></td>
<td>35 min</td>
</tr>
<tr>
<td><strong>Temperature</strong></td>
<td>Water-cooled plate</td>
</tr>
</tbody>
</table>

Table 3.5: Parameter for preparation of IrAu TES

TES structure has been done after the evaporation by means of positive photolithography. To remove the part in excess of the film, Plasma Etching was used. In table 3.4 the parameter used for sensor structure are showed.

The superconducting transition has been measured with a current of 0.49 μA and the critical temperature was found to be 83.2 mK, the normal state resistance was about 2.5 Ω and the residual resistance in the superconducting state was about 38 mΩ; the width of the transition was 0.5 mK.

3.6.2 Dilute magnetic alloys

[73] The use of dilute alloys as system for TES sensors has been adopted by different groups [74] [75]. In this case magnetic impurities are used to suppress the transition temperature of pure metals.

In the work of Deiker et al. [74], the characterization of Al:Mn TES has been presented. The films were prepared by co-sputtering from two target one of Al-Mn alloy (99.7 % Al, 0.3 % Mn) and the other of pure aluminum. The sensors obtained show in-band noise consistent with the theory, with a noise equivalent power of $7.5x10^{-18} W/\sqrt{Hz}$. The heat capacity is not increased by the presence of magnetic impurities in concentration of few thousand ppm and these sensors are weakly sensitive to small external magnetic field.

This method of evaporation allows to increase the concentration of manganese contained in the film respect to the one permitted by the Mn
Figure 3.14: Comparison between the transition of Zn:Mn film prepared with thermal evaporation at different currents

solubility in Al, that at room temperature is only of 0.002%.

In the work of B. Young [75] the transition temperature was adjusted using ion implantation on superconducting films directly, or by in situ magnetic ion doping using a doped single target or two single element targets co-sputtered.

Our films were prepared using thermal evaporation of the suitable dilute alloy target. The bulk alloy was prepared by Prof. Andrea Palenzona and Dr. Pietro Manfrinetti of the Department of Chemistry (University of Genoa), by melting the wanted ratio of pure metal.

The first alloy we tested was the Al:Mn and three different manganese concentrations bulk were prepared: Al$_{0.998}$Mn$_{0.002}$ Al$_{0.996}$Mn$_{0.004}$ and the super-saturated Al$_{0.994}$Mn$_{0.006}$. Different concentration were prepared to test how the critical temperature decreases following the manganese concentration and therefore to compare the results with the curve calculated in [?]. With the super-saturated alloy we expected a transition temperature of about 100-150 mK, but for the evaporated films we didn’t achieve values below 200 mK, showing a sort of saturation. The main drawback of our method was the impossibility to increase in a controlled way the concentration of magnetic ions in the superconducting film since we were limitated in the bulk alloy preparation by the maximum solubility
of manganese in aluminum that is 0.62 % at about 650°C.

The second system investigated was Zn:Mn, the advantage offered by this alloy is the theoretical possibility to decrease the critical temperature to 100 mK as it is derived by the experimental law in [?]. The bulk alloy has been prepared following the recipe in [77], the bulk with 25 ppm of manganese was prepared in order to have 100 mK as transition temperature; higher manganese concentration may destroy the superconductivity. The sensors were prepared by means of thermal evaporation, small lumps of the zinc alloy were positioned in a tungsten basket. The adhesion of zinc film to silicon substrate is not good, therefore it was necessary to evaporate before the dilute magnetic alloy, a stick layer of silver of thickness of 30-50 Å (evaporated from molybdenum boat). The Zn:Mn film was evaporated at a rate of 23-30 Å/s and the final thickness was around 900 Å.

The expected transition temperature was around 100 mk, but we measured about 250 mk and 500 mK, respectively for the bulk and the film. We have also noted a very large transition width. These results disagree with our expectation based on reference [?]. The reason has not yet been identified; test on more concentrated alloys are necessary. In particular we are testing a Mn concentration of 50 ppm, twice the previous, in order to verify the law of transition temperature vs. the Mn concentration the effect of distillation in the film growth by thermal evaporation. However this result are considered encouraging since for Zn-Mn system we can increase the Mn concentration until 0.5 % at 419°C for further lowering the transition temperature.

3.7 Magnetic sensors

The improvements in the energy resolution obtained by magnetic calorimeters in this last decade is impressive. During the last conference on Low Temperature Detectors (LTD10 - Genoa 7-11 July 2003) A. Fleischmann et al. [32] presented the very nice result of 3.4 eV of energy resolution on $^{55}Fe$ $K_\alpha$ (resolving $K_{\alpha 1}$ and $K_{\alpha 2}$) and $K_\beta$; the particular of the spectrum with the resolution of $K_{\alpha 1}$ and $K_{\alpha 2}$ is shown in Fig. 3.14.

These sensors have been considered as a possible upgrade for MANU2 experiment since their resolution and their stability for long measurements make them promising.

In the next section I will describe the principles of magnetic calorimeter while the possible application for the rhenium experiment will be described in chapter 8.
Figure 3.15: $^{55}Fe$ energy spectrum obtained with a magnetic calorimeter by the group of Heidelberg [32]. The energy resolution is 3.4 eV at 5.9 keV.

### 3.7.1 Principles and theory of operation

A magnetic sensor consists in a dilute concentration of paramagnetic ions in a metallic host set in a weak magnetic field; the system studied and developed in Heidelberg is metallic gold with 300 ppm of isotopically enriched $^{166}Er$. The typical shape of the sensor is a cylinder, a laser-cut disc of 50 μm diameter and 25 μm high. The absorber is a gold foil with the wanted dimensions in order to have a suitable stopping power. The magnetic field used to polarize the Er magnetic moment is set at 3 mT. As for all the microcalorimeters, the released energy increases the temperature of the detector. Here, instead of a resistance $R(T)$, like in the TES sensors, a magnetization $M(T)$ drops down with the impulsive increase of temperature. For an absorbed energy $\delta E$ in a volume $V$, the corresponding change in magnetic moment is:

$$\delta M = \frac{\partial M}{\partial T} \delta T = \frac{\partial M}{\partial T} \frac{\delta E}{C}$$  \hspace{1cm} (3.4)
the amplitude output signal is modulated by the characteristic magnetization vs temperature. The magnetization signal is read by the input coil of low noise SQUID magnetometer. In particular, a two stage SQUID read out system is used: a first detector SQUID in which the input coil is directly near the sensor, and a second SQUID as voltage amplifier. This configuration allows a lower noise level respect to a single stage SQUID system.

3.7.2 Optimization of magnetic calorimeters

It is important to note that, unlike TES sensors, where excess noise shows how the principles ruling the electrothermal response are not yet clear, magnetic calorimeters are based on a well known thermodynamic system and therefore it is possible to have a reliable prediction of the detector performances [78]. The sensitivity of these detectors, calculated as the ratio between the change in magnetic flux following the release of energy and the energy itself, has been estimated as a function of a number of parameters:

\[
S = S(C_a, g, \alpha, T, x, r, h, B) = \frac{\delta \Phi}{\delta E} \tag{3.5}
\]
where \( C_a \) is the heat capacity of the absorber, the g-factor of the paramagnetic ions, \( \alpha \) the strength of RKKY (Rudermann, Kittel, Kasuya and Yosida) exchange interaction among spins mediated by the conduction electrons, \( T \) is the operating temperature, \( x \) is the concentration of ions, \( r \) and \( h \) are the radius and the thickness of the cylindrically shaped sensor and \( B \) the applied magnetic field.

In order to optimize the performances of the magnetic calorimeters the sensitivity must be maximized. The magnetic flux variation may be written as:

\[
\delta \Phi = \mu_0 G \frac{1}{r} V \frac{\partial M}{\partial T} \frac{1}{V_c + C_a} \delta E
\]  

(3.6)

where \( \mu_0 \) is the vacuum permeability, \( G \) e geometrical factor, \( V \) the sensor volume and \( c \) is \( C_{\text{sensor}}/V \) which takes in account the parameters \( \alpha, g, x,T \) and \( B \). Therefore the sensitivity is given by:

\[
S = \frac{\delta \Phi}{\delta E} = \mu_0 G \frac{1}{r} V \frac{\partial M}{\partial T} \frac{1}{V_c + C_a}
\]

(3.7)
To maximize this quantity is useful to use other parameters, writing $S$ as a function of $\xi = 2r/h$:

$$S = \mu_0 G(\xi) \left(\frac{2\pi}{\xi}\right)^{\frac{1}{3}} \frac{\partial M}{\partial T} V^{2/3} V c + C_a$$  \hspace{1cm} (3.8)

The optimization of the sensor dimension follows the maximization of the factor $G(\xi) (2\pi/\xi)$. This has been done by means of finite elements simulation with the software FEMM; in doing this the gap between the sensor and the SQUID loop has been taken in consideration. The result is that the optimal proportion between sensor radius and thickness is:

$$h_{opt} = 0.25 r_{opt}$$

The optimal sensor volume is obtained by maximizing the factor $V^{2/3}/(V c + C_a)$ in relation to the absorber heat capacity:

$$c V_{opt} = 2 C_a$$

Substituting the $V_{opt}$ as a function of the absorber heat capacity, the maximum sensitivity is given by:

$$S_{max} = \mu_0 G(\xi_{opt}) \left(\frac{2\pi}{\xi_{opt}}\right)^{\frac{1}{3}} \frac{\partial M}{\partial T} 2^{2/3} 3^{1/3} c^{-2/3} C_a^{-1/3}$$  \hspace{1cm} (3.9)

or, extracting the other parameters dependence [78]:

$$S_{max} = 1.7 \times 10^{-9} \left(\frac{g}{C_a \alpha T^2}\right)^{1/3} \Phi_0/eV$$  \hspace{1cm} (3.10)

where $\Phi_0 = 2.07 \times 10^{-15}$ Wb is the flux quantum. The other parameters not present in the previous relation can be calculated as a function of $C_a$, $g$, $\alpha$ and $T$.

Regarding the magnetic sensor intrinsic sources of noise, they are mainly the SQUID noise, the thermodynamic fluctuations and the magnetic Johnson noise due to fluctuating current in conductors in the vicinity of the pick up coil and SQUID. In [32] the white noise level was of 1.4 $\mu\Phi_0/\sqrt{Hz}$ in which the SQUID noise was estimated as 1.1 $\mu\Phi_0/\sqrt{Hz}$ and the magnetic Johnson noise of the sensor 0.9 $\mu\Phi_0/\sqrt{Hz}$. There is still a not completely understood $1/f$ component that seems to be associated with the presence of spins in the sensors.

In the end, the use of magnetic calorimeters for x-ray and particles detection shows interesting properties, the first is the energy resolution which
is expected to reach values of $\delta E/E$ better than $3 \times 10^{-4}$, then the possibility to use large mass absorbers would permit to have high quantum efficiency. In particular for the rhenium experiment this will strongly reduces the number of channel necessary to have the suitable count rate. In the end the compatibility with microfabrication technique which can lead to multiplexing schemes has been studied.
Chapter 4
Microcalorimeter energy absorber

4.1 Introduction
The absorber converts the incident energy into heat which consequently increases the temperature of the absorber itself that is transformed in a signal in the sensor. The absorber material and its dimension have to be optimized for the specific applications. A first important parameter to evaluate is the heat capacity since we have seen that the energy resolution is proportional to the square root of C and the temperature excursion is proportional to 1/C. The absorber must also have a high absorption efficiency in the energy range of interest, so it is important to find a just compromise between the low heat capacity and the stopping power. The low temperature heat capacity for metal is dominated by the electron contribution given by:

\[ c_{el}^V = \frac{n\gamma T}{N_A} \]  

(4.1)

where \( N_A \) is Avogadro’s number, \( \gamma \) the Sommerfeld parameter and \( n \) is the atomic concentration. For superconducting material at a temperature below the critical one, the electric heat capacity is decreased due to the condensation of electrons in Cooper pair and it has the form:

\[ c_{el}^V(sup) = \alpha e^{\frac{\beta T}{T_c}} \]  

(4.2)

where \( \alpha \) and \( \beta \) are parameters depending from the superconductor; the main contribution is the given by the lattice heat capacity:

\[ c_{ph}^V = \frac{12\pi^4}{5} n k_B \left( \frac{T}{\theta_D} \right)^3 \]  

(4.3)
For dielectric materials the heat capacity has only the phonon contribution, since there are not free electrons. The absorption efficiency for photons is determined by the knowledge of the absorption length $\lambda$ expressed in $g/cm^2$ ($\lambda = 1/\mu/\rho$ with $\mu$ is the mean free path and $\rho$ is the density; it can be extracted for different material from specific curves) and inserting it into the relation:

$$I = I_0 e^{-t/\lambda}$$  \hspace{1cm} (4.4)

where $I$ is the intensity remaining after traversal of thickness $t$ expressed in mass/unit area, and $I_0$ is the initial intensity [79]. For massive particles as electrons or alpha particles it is important the range of the in the absorber before they are absorbed [79]. The absorber is then sized for the absorption of the whole energy of the radiation in a specific range of values. In fact, the heat capacity is also calculated in order to allow the detection of the lower and the upper energy value of the range without loss of performances. It has to be small enough to give a measurable signal for the low energy events but not too small to have resistance variation bigger then the superconducting transition for the higher energy events. So the heat capacity can not be smaller then:

$$C = \frac{E_{\text{max}}}{\Delta T_{\text{max}}} = \frac{E_{\text{max}} \langle R \rangle}{\langle T \rangle \Delta R_{\text{max}}} = 0.63 \frac{\alpha E_{\text{max}}}{T_C}$$  \hspace{1cm} (4.5)

in order to keep the pulse to detect from 10 % and 90 % of the transition.

In the next section I will describe the main features of dielectric and superconducting absorbers, in the last section will discuss the importance of having the radioactive source inside the absorber itself when it is important to analyze a radioisotope spectrum in details.

### 4.2 Dielectric absorber

As described in the previous section, the heat capacity of dielectric absorber seems to be ideal for a microcalorimeter absorber, since the main contribution is due to the lattice, the same is also valid for semiconductor material, in which the electric contribution is negligible. Nowadays no many experiments use this type of absorber, in fact there are some drawbacks whose the main is the fact that the excitations created after the release of energy, electron-hole pair, have usually a recombination time longer then the pulse formation. The thermalization
efficiency is defined as the ratio between the heat amount and released energy and can be written as:

$$\eta = \frac{E_{\text{gap}} + \Omega}{2E_{\text{gap}} + \Omega}$$

(4.6)

where $E_{\text{gap}}$ is the energy gap and $\Omega$ is the fraction that goes in the phonon channel.

At very low temperature, the creation of long-lived metastable states, composed by hole-electron couples, subtract energy to the phonon system causing an excess noise, which is proportional to the $\sqrt{FW}$, where $w$ is the primary energy for exciting an e-h couple. In this frame it is expected a noise higher than the thermodynamical one, and an energy resolution varying with the $\sqrt{E}$. A clear evidence of this effects has been seen in BeO absorber in which the energy resolution of a peak at 112 eV was found 8 times higher than the baseline energy equivalent resolution, and well fitted by a binomial function [82].

An interesting case in which dielectric absorbers are used is the Milano neutrino mass experiment (MIBETA) Since the aim of this experiment is the estimation of neutrino mass upper limit by means of the analysis of $^{187}$Re beta spectrum, the absorber must contain the radioactive atoms inside and therefore be a compound containing rhenium. The dielectric compound that they have chosen is $A_{\text{g}}ReO_4$ and its main feature are described in [23]. Besides the very good results they obtained, there are still unexplained behavior of the absorber. In particular the fact that all x-ray peaks coming from external calibration sources show a not negligible tails on the low energy side. They succeeded in reproducing that shape fitting the peaks with two gaussian functions with the same width. Around 2.4 keV the two gaussians are separated by 37 eV and the area ratio between them is 3.4 %. They claim that a possible reason for the asymmetric shape of external source x-ray peak, is a deterioration of the crystal surface which lead to a different thermal response between the events happening on the surface and the ones happening in the inner part of the crystal. In order to understand this behavior a future run with an external source able to excite the Re K-edge at 71.7 keV is in progress. Another point that seems related to the dielectric absorber characteristics is the energy resolution dependence with the energy.

### 4.3 Superconducting absorber

The main contribution of heat capacity of a superconductor for temperature below the transition (in order to have few free electrons), is the
lattice one therefore suitable for obtaining good energy resolution. The energy release inside a superconductor leads this to a state far from the equilibrium in which may Cooper pair are broken and quasiparticles created. The model describing this situation was developed by Kaplan [80] and he calculated that the time which is needed to the quasiparticles to recombine in superconductors at a temperature of 100 mK is several seconds. A more refined analysis has been done by Kozorezov [81]. If this time is compared to the pulse formation time, the first conclusion is that superconducting materials are not suitable as energy absorber. Besides that, superconductors are used and very good performance are obtained. The group of Genoa [83] explored the response to radiation of several superconducting metal. were tested as bolometer absorber and the efficiency measured as a function of temperature. The empirical efficiency was evaluated comparing the integrated area of the averaged pulse, coming from the interaction of $\alpha$ particles in the absorber, with that of the calibration thermal pulse generated by a resistive heater and corresponding to the same energy of the selected $\alpha$ particles. The result was that for each superconductor tested, the efficiency sharply decreases for temperatures below $2 \times 10^{-4} \theta_D$. This behavior was shown for several first type superconductor such as Re, Al, In, Zn and also for second type as Nb.

The reason for this experimental behavior is not yet theoretically understood, but what is clear is that superconducting absorber could be used as low temperature microcalorimeter absorbers and, from the recent results, they are giving very good results in term energy resolution.

### 4.4 Internal radioactive source

Microcalorimeters are usually developed for high resolution x-ray measurements, devoted mainly to astrophysical investigations and material science, therefore the detected radiations comes from an external source (even extremely far). In experiments where it is very important measure all the energy released during the radioactive decay, including the energy taken by the atomic relaxation, an absorber with inner radioactive source is suitable since all the energetic events corresponding to all the de-excitation processes, take place inside the absorber itself.

Considering $\beta$ decays, the final electron spectrum has not the typical shape expected for a transition between two defined isobar levels, but the presence of several atomic and molecular final states, each of them
leading to a different end point energy for the emitted electron, modifies the final part of the spectrum. In fact the number of electrons is proportional to:

\[ N(E) \propto \sum_i W_i (E_{0i} - E) \sqrt{(E_{0i} - E)^2 - m^2_\nu} \]  

(4.7)

where \( \epsilon_i \) and \( W_i \) are the energy and decay probability of the excited state \( i \). Consequently the Kurie plot results distorted in the final part simulating a possible negative \( m^2_\nu \) [58].

The problem of the final state in determining the total energy available for the decay may be solved extrapolating from the final spectrum any possible excited state contribution, but this way depends too much theoretical model for atomic de-excitation. In a \( \beta \) decay the energy available is
shared among the electron neutrino (not detectable), the electron end the atomic de-excitation energy, therefore in order to measure the neutrino complementary spectrum the electron energy and the atomic energy are to be measured simultaneously. In the obtained spectrum, called calorimetric spectrum, the total energy released without the neutrino one is detected. Therefore:

\[ N(E) \propto (Q - E)\sqrt{(Q - E)^2 - m_\nu^2} \]  

(4.9)

where \( Q \), the decay end-point energy and correspond to:

\[ Q = E_{-01} + \epsilon_1 = ... = E_{0i} + \epsilon_i \]  

(4.10)

The end point region of a calorimetric beta spectrum is therefore the same expected for a transition between defined initial and final states. To obtain such a spectrum it is necessary that atomic de-excitation energy is detected with the same efficiency of the emitted electron and that the element processes in the detector following the interaction have low energy activation in order to detect the small atomic (or molecular) de-excitations. This is the indication that the radioactive isotope have to be contained inside the sensible part of the detector. This solution permit also to forget about the corrections due to the interaction of the radioactive isotope with envelope.

The fact of having an inner source is extremely important in the case of neutrino mass determination since small deviation in the final part of the spectrum can lead to unreal quantity determinations. At the moment the two rhenium experiment running use absorber with \(^{187}\text{Re}\) atoms directly inside, the Genoa group as natural metallic rhenium (containing 65%), the Milan group as \( \text{AgReO}_4 \).

Not only for neutrino mass experiment, but also for each experiment which aim to measure spectra with high resolution especially at low energy.

To have the radioactive source uniformly dispersed inside the absorber, is not always so easy a task, I developed a method to obtain superconducting absorber containing the isotope tested on Rare Earths metal but suitable for a large number of case. This method was study above all to measure the Electron Capture spectrum of \(^{163}\text{Ho}\) [27], interesting for the determination of energy levels determination [26].
Chapter 5

Development of 163-Ho superconducting absorber

5.1 Introduction

As described in the first chapter, a detailed analysis of $^{163}$Ho Electron Capture spectrum would be useful to solve the problem of the red-shift of x-ray lines that have been measured. From the experiments already done, it is evident how difficult would be to improve the detector performances to reach the suitable energy resolution.

One of the best solution would be the use of low temperature detector with inner source absorber, method already tested by the Genoa group [26] but in not with the source homogeneously dispersed. In fact the solution of preparing a superconducting absorber with the $^{163}$Ho atoms homogeneously dispersed is not straightforward when the radioactive source is in liquid form. The problem arises by the thermo-electrical properties of rare earths metals which make the reduction (passage from the ion to atom) of these element very difficult above all when the amount of material to treat correspond to liquid drops with few microlitres volume. In the next chapter I will better explain the rare earths reduction problem and describe the solution for the production of superconducting absorber in which the radioactive atoms are really well dispersed inside.

5.2 Thermoreduction of rare earths

Metallothermic methodologies for reduction of rare earths oxides or salts are typically used [84], [85] and [86] to prepare samples of metal rare earths as containing radioactive isotopes. The amount of lanthanons
used in these reactions is in the range between tens of milligrams and several grams. In particular the reduction of holmium has been studied carefully and a suitable method has been developed; the same method may be used for other radioisotopes which need to be homogeneously dispersed in a metal host.

In order to analyze the efficiency of the method, we decided to use a particular holmium isotope whose emitted particle could be detected easy. The suitable candidate is $^{166}\text{m} \text{Ho}$, a beta-decaying nucleus with half-life of $1.2 \times 10^3$ years; its spectrum is composed by a continuum electron spectrum, whose end-point is at the energy of 60 keV, and by several gamma lines, the most intense are at 184 keV, 280 keV, 711 keV and 810 keV. In particular, the line at 184 keV has 100% branching ratio, so one can estimate the amount of radioactive atoms just by analyzing this one. The result I reached is the possibility to obtain a sample of tin alloy and tin metal containing radioactive $^{166}\text{m} \text{Ho}$ in intermetallic form.

In the first experiment, the amount of holmium to be reduced was about 0.02 $\mu$g corresponding to an activity of 1110 Bq (I’ve used only holmium coming from $^{166}\text{m} \text{Ho}$ radioactive standard solution without natural holmium as carrier). Such an amount of rare earths is impossible to deal with common methods of reduction because it’s easy to lose in the reaction vessel. The procedure to prevent this, may be divided into two steps: in the first step the inorganic salt is reduced by thermoreduction with yttrium and/or scandium in a tantalum crucible, heated in an oven; the second step is the reaction of the holmium metal and the residual yttrium and/or scandium metal, with a large amount of tin. This last reaction had to be made in the same crucible and the time occurring to open the crucible, to fill it with tin and to close was minimized in order to make negligible the re-oxidation of the holmium.

5.3 Step I: thermo-reduction of holmium

The reduction of Rare Earths has always been a problem, many difficulties come from the low electrochemical potential and from the high stability of their inorganic compound, mainly their oxide.

The first feature makes quite impossible the elettrochemical reduction using conventional cathodes. The method proposed in [87] in which the reduction of several Rare Earths has been obtained using an electrochemical cell with an amalgam cathode, was not successful in reducing holmium. Following this method I try with a different amalgam cathode in which the mercury was mixed with gallium [88], each metal has its
task: Mercury is important for making difficult the reduction of hydrogen and gallium has to bind holmium in intermetallic compound when the reduction happens. With this method I found a particular compound containing mercury, holmium and gallium. Another compound containing holmium and gallium was obtained using a cathode of pure gallium. This two kind of compounds were not useful to prepare an absorber for the measurement of $^{163}$Ho electron-capture spectrum.

The stability of Rare Earths inorganic compounds makes difficult the thermoreduction, above all when very small amount of material are considered because it’s easy to lose during the reaction.

We decided to follow the principles of thermoreduction procedure which allows to obtain, with the help of high temperature, the metal in its elemental form, starting from inorganic compound such as salts or oxide. In this reaction the oxygen or the anions are captured by another metal used as reductant (denoted dy M, with valence x). The general reaction describing the thermoreduction involving trivalent Rare Earths compounds can be written:

$$xRE_2O_3 + 6M \rightarrow M_2O_x + 2xRE$$ (5.1)

In order to have the equilibrium shifted to the right, the metal M must have a more stable oxide, i.e. a higher tendency to bind oxygen.

Analog reactions can be written for other Rare Earths compounds. Looking for the reduction of holmium, this reaction becomes:

$$xHo_2O_3 + 6M \rightarrow M_2O_x + 2xHo$$ (5.2)

The parameter that helps in finding the the suitable element to use as reductant, is the Gibbs Free Energy (GFE) of oxide formation [89]: the higher is its absolute value the higher is the stability of the oxide. The value of GFE for some elements’ oxide are here listen:

$$Ho_2O_3 : \Delta G = -1791.1 \text{ KJ/mol}$$ (5.3)
$$CaO : \Delta G = -603.3 \text{ KJ/mol}$$ (5.4)
$$Al_2O_3 : \Delta G = -1582.3 \text{ KJ/mol}$$ (5.5)
$$La_2O_3 : \Delta G = -1705.8 \text{ KJ/mol}$$ (5.6)
$$Dy_2O_3 : \Delta G = -1771.5 \text{ KJ/mol}$$ (5.7)
$$Er_2O_3 : \Delta G = -1808.7 \text{ KJ/mol}$$ (5.8)
$$Sc_2O_3 : \Delta G = -1819.4 \text{ KJ/mol}$$ (5.9)
$Y_2O_3 : \Delta G = -1816.6 \, KJ/mol$ \hspace{1cm} (5.10)

Another useful note is to check how the Gibbs Free Energy changes with the temperature, so using the parameters given in [89] we can compare the behavior of several elements:

![Figure 5.1: Gibbs Free Energy for Lanthanum, yttrium and scandium](image)

The law that fit this point is:

$$\Delta G = A + BT + CT \log T$$ \hspace{1cm} (5.11)

and we can see how the trend is quite linear and the curves are not crossing each other, so we can say that if a compound is more stable at room temperature than the holmium compound, so it will be at high temperature too. In order to have the best conditions to reduce the holmium oxide, the metals we could use as reductant would only be yttrium or scandium (with the exception of actinides).

### 5.3.1 Experimental consideration

Generally, the quantity of radioactive source one has to deal with is very small, so that it is extremely important to avoid losses during the whole procedure and to have the higher possible efficiency in collecting the final metal. For these reasons, all reactions must take place in a sealed container. After deciding the total amount of radioactive source to be
reduced (in order to obtain a proper activity in the final sample), the corresponding drop has to be dried into the crucible, on its bottom (this procedure has to be carried out at room temperature or just few degrees above, in order to prevent evaporation of the radioactive isotope too). The aim of this experiment is to have a very small amount of metal radioactive holmium spread in tin bulk. The procedure to be performed needs to involve only tens of nanograms of compound to be reduced, and needs to be the most efficient as possible in order to collect nearly the whole amount of radioactive isotope.

The radioactive source is 1 ml $^{166}Ho$ source in form of $HoCl_3$ in acid solution of HCl 0.1 M, the nominal activity is $1 \mu Ci$

In order to make the minimum number of treatments, the best thing to do was to evaporate few microliters of this solution directly on the bottom of a Tantalum crucible (inner diameter 0.8 cm, high about 4.5 cm), in this case 30 $\mu l$ were evaporated for an activity of 1110 Bq.

The amount of inorganic compounds on the bottom of the crucible is estimated to be:

\[
HoCl_3 \rightarrow 0.02 \mu g \text{ or } 10^{-10} mol
\]

\[
HCl \rightarrow 110 \mu g \text{ or } 3 \times 10^{-6} mol
\]

The fact that we have chloride instead of oxide is not a big problem because the stability of these salt with scandium and yttrium is high as well therefore they are able to gather the chlorine from the holmium. The second part of the reduction step consists in weighing the right amount of reductant metal and mixing it with the oxide/material in the crucible. The metal has to be in excess, so that it can gather not only the oxygen bound to the radioactive isotopes, but also all the other anions coming from the dried source (such as the anions of the acid in which the isotope was solved) and all impurities contained in the crucible. The amount of reductants used has been:

\[
Y \rightarrow 0.1020 g
\]

\[
Sc \rightarrow 0.0230 g
\]

After this step, the crucible is sealed by arc welding under a pure argon atmosphere, then it is closed again in a quartz ampoule and set in a high temperature furnace for. The temperature was increased to 300°C for one night and then increased to 900°C for three days. After this lapse
of time the reduction process is supposed to be complete; what we can expect to find inside the crucible is the reductant metal, both forming inorganic compound with oxygen and other anions and in metal form, as well as the radioactive material reduced by M.

This is the end of the first step; how can we see if some reduction has taken place? The method to know this is to consider that if some holmium atoms have been reduced, the holmium metal has to be in equilibrium with its vapor, that is some holmium must be in vapor phase (the amount depends on the temperature) and when the temperature decreases this vapor has to condense on the wall of the crucible.

We can estimate how much holmium has to be on the cap looking at the vapor pressure vs. temperature. The holmium vapor pressure at 1173 K is about 4.6 $10^{-8}$ atm, the volume inside the crucible is 2.26 cm$^3$, thinking of holmium vapor as a perfect gas, we can calculate the number of mol inside.

Using the relation:
\[ n = \frac{PV}{RT} \]  
we obtain for the values given above:
\[ n(1173 \text{ K}) = 1.67^{-12} \text{ mol} \]  
In the same way the number of mols corresponding to the temperature of 300 K can be calculated:
\[ n(300 \text{ K}) = 9.17^{-14} \text{ mol} \]

In first approximation the holmium deposited is the difference in mols between the two value above, from this we can calculate the activity and the specific activity.

\[ \Delta n = 9.88^{-13} \text{ mol} \]
\[ A = 15.7 \text{ Bq} \]
\[ A_S = 1.33 \text{ Bq/cm}^2 \]

Now we can calculate the activity expected from the cap:
\[ A_C = 0.67 \text{ Bq} \]

So when we open the crucible we can find deposited on the cap some $^{166}\text{Ho}$. The figure shows what we have measured with a gamma detector,
Figure 5.2: Vapor pressure curves
Figure 5.3: Spectrum showing the presence of $^{166m}$Ho on the cap a scintillator NaI(Tl) when we put on it the Tantalum cap.

In particular if we subtract the background from this spectrum and fit the peak corresponding at 184 KeV, calculate the area being careful to made the suitable correction due to the geometry and to the absorption of radiation in the media between the source and the detector, we can measure the activity and to go back to the number of atoms, since the branching ratio for this transition is 100%.

The parameters coming from the analysis fit the equation:

$$f(x) = g + p0$$  \hspace{1cm} (5.23)

$$g = P1 e^{-\frac{(x-P2)^2}{2P3^2}}$$  \hspace{1cm} (5.24)

$$p0 = P4$$  \hspace{1cm} (5.25)
Calculating the area under this curve and subtracting the area under $p_0$ we can estimate the activity of 0.60 Bq.
This value, comparing to the theoretical one, shows that the reduction have taken place. So the holmium around the crucible is around 15 Bq, corresponding to 1/100 of the total, the larger part of this will be recovered by the tin when it is heated and melted, in fact the volume of this metal will be a little smaller than the volume of the crucible.
In this case we can estimate that about 1 Bq will be lost on the wall of the crucible, that is about 1/1000 of the total amount of holmium.

5.4 Step II: tin Alloys

At the end of the first step, when we open the crucible, we don’t still know if the reduction have taken place, but if this happened we can collect the holmium reduced together the other metals, in intermetallic compound with tin.
To estimate the amount of tin to be melted, we have to think what intermetallic we need; in this case we want just demonstrate that is possible to have the Rare Earth spread in a uniform way in a tin intermetallic compound.
Having use yttrium as the most abundant reductant we can consider the $RESn_3$ compounds, these are the same for yttrium and holmium, so the amount of tin to use would be 75.6 weight %.
Another important think to look after is the final activity per milligram of the sample. So the amount of tin as to be more than the 75.6 weight % and we chose a final activity around 1 Bq/mm$^3$, thinking that all the holmium will be in the final bulk. To have 1 Bq/mm$^3$ we had to use about 6.4 g of tin, but it was too much to be contained in the crucible, so we used 4.9180 g that corresponds to 97 weight % of the element contained in the crucible. The activity in these conditions will be about 0.22 Bq/mg always if all the holmium is in the final bulk (tin and intermetallic).
The procedures to obtain this intermetallics are described in [90].
After having close for the second time the same crucible by arc welding, the metals were melted in a high frequency induction furnace in four steps, increasing the temperature till 1200°C (500°C → 700°C → 900°C → 1200°C). In order to have the formation of crystals of the Rare Earths intermetallic, we need to annealing the sample for several days (15 days) at the temperature of 400°C, below the $RESn_3$ decomposition temperature.
After these heating treatments the sample was extracted and an internal slide was cut. This was polished and lapped, what we saw after these procedures, was the presence of macroscopic intermetallic crystals.

In fig. 5.4 we can see a particular of the intermetallic crystal, in the center of there is the primary formation with around the second formation, coming from the action of liquid tin on the present crystals. The first kind of intermetallic compound that appears when the temperature cools down from the temperature where we have only liquid phase, is $RESn_2$, then annealing at the temperature just under the temperature of decomposition of the compound of interest ($RESn_3$), the tin works on the crystals, destroying the compound present and making the new one.

![Image of intermetallic crystals in tin](image)

**Figure 5.4: Image of intermetallic crystals in tin**

### 5.5 Analysis of the activity

The reaction between tin and the metals in the crucible have taken place, but what we are interest in if inside the bulk there would be the holmium...
too. In order to see this and to estimate how it is located, several measurements of activity were made. First of all the activity of the crucible and of the dust, this shows an amount holmium about 90 Bq, this corresponds to the holmium lost, the 8% of the total.

The second measurement made is the activity of the slide, a first run as soon as the slide was cut, a second run after having polished the oxidized surface around it. In this way we can check if the holmium is only on the surface or not. Comparing the spectra acquired what we see is that the activity is unchanged in a remarkable way, so we can say that the holmium is totally contained in the inner part of the bulk.

Another measurement was done in order to check if there was a gradient in concentration, so two spectra were acquired exposing the two sides of the slide. If the concentration was not homogeneous, what we had to see, would be a different activity between the two spectra. The result of this test was that no differences were found between the two spectra, so we can say that the holmium is spread inside the bulk in uniform way.

Figure 5.5: Spectrum from the slide
The activity we can estimate from the analysis of 184 KeV energy peak is 170 Bq.
The measurement of the specific activity was made taking a sample from the center of the slide, weighting it and acquiring its spectrum. The weight of the sample is:

\[ P = (0.2323 \pm 0.0001) \ g \]  \hspace{1cm} (5.26)

The activity was estimated always from the 184 KeV peak, subtracting from the area under the experimental points, the area under the line that is the background, and then normalizing for the live time of the acquisition. The fit of 184 KeV peak with the function:

\[ f(x) = g + p1 \]  \hspace{1cm} (5.27)

that is a gaussian and a polynomial of degree 1. From this fit we can find where the peak is centered, the sigma and the function for the background B.

\[ B(x) = 2x + 723 \]  \hspace{1cm} (5.28)

The area under the peak, calculated between bin 400-480, is:

\[ A = (5893 \pm 7) \times 10^2 \]  \hspace{1cm} (5.29)

the background calculated as the area under B(x) between the same bin, is:

\[ B = (1282 \pm 3) \times 10^2 \]  \hspace{1cm} (5.30)

So the area corresponding to the \( \gamma \)-rays acquired is the difference between the two values:

\[ C = A - B = (4611 \pm 16) \times 10^2 \]  \hspace{1cm} (5.31)

This value, normalized for the live time, that is 43200 s, gives the number of \( \gamma \)-rays. The activity is calculated considering the whole solid angle, the self-absorption through the tin and the efficiency of the detector. After these corrections the activity estimated is:

\[ \beta = (46 \pm 1) \ Bq \]  \hspace{1cm} (5.32)

Now we can calculate the specific activity:
\[ \beta_S = 46 : 232.3 = 0.20 \text{ Bq/mg} \rightarrow (0.20 \pm 0.01) \text{ Bq} \quad (5.33) \]

The total amount of material melted in the crucible was:

\[ Sn \rightarrow (4.918 \pm 0.001) \text{ g} \quad (5.34) \]

\[ Y + Sc \rightarrow (0.125 \pm 0.001) \text{ g} \quad (5.35) \]

That is 5043 mg.

With this value we can calculate the total activity in the bulk:

\[ \beta_B = 0.20 \times 5043 = 1010 \text{ Bq} \rightarrow (101 \pm 5) \times 10 \text{ Bq} \quad (5.36) \]

The missing activity may be found inside the crucible and in the residual powders. So an analog measurement was made for one of the external pieces cut from the slide, the activity we measured (using the same method as before) was always of 0.20 Bq. After this confirmation, we can say that, considering samples of hundreds milligrams weight, the holmium is equally distributed.

Furthermore it is possible to compare the activity of the intermetallic with the activity of pure tin. So we cut a sample of 600x600x250 \( \mu \text{m}^3 \), corresponding to 0.66 mg. We acquired the spectrum from this small piece, the presence of holmium this time was not so clear, but subtracting from this the spectrum with the background and fitting the 184 KeV peak with the same function:

\[ F(x) = g + p0 \quad (5.37) \]

we could find the evidence of holmium.

In this case as well, we calculated the activity and the specific activity:

\[ \beta = (0.061 \pm 0.010) \text{ Bq} \quad (5.38) \]

\[ \beta_S = (0.09 \pm 0.01) \text{ Bq/mg} \quad (5.39) \]

With this value we can now make some considerations. The bulk, as we say, is made of tin with some intermetallic crystals inside, the concentration of holmium may be different if we have a sample of tin only or a sample of tin containing slides of intermetallic. The previous calculations give us the activity in tin, so, using the activity of the center of the slide (it contains tin and intermetallic) and knowing the percentage of intermetallic compound, we can see if the considerations on two different
activities are correct. We calculated that the specific activity of the bulk is 0.20 Bq. Considering the center its weight is 232.3 mg the fraction of weight corresponding to the intermetallic is:

\[ P_I = 0.143(ScSn_2) + 0.522(YSn_3) = 0.665 \rightarrow (0.665 \pm 0.001) \mathrm{g} \quad (5.40) \]

We use the approximation that the yttrium is present only in \( YSn_3 \) and scandium only in \( ScSn_2 \), so the relation is:

\[ P_{I/T} = 13\% \quad (5.41) \]

We derive the two weights:

\[ W_I = (30 \pm 1) \mathrm{mg} \quad (5.42) \]
\[ W_T = (202 \pm 1) \mathrm{mg} \quad (5.43) \]

The activity coming from the holmium contained in tin is:
Figure 5.7: Fit of 184 KeV peak for the 600*600*250 $\mu$m$^3$ sample

$$\beta_T = 202 \times 0.09 = 18 \text{ Bq} \to (18 \pm 2) \text{ Bq} \quad (5.44)$$

Subtracting this from the total activity coming from the center we can obtain the number of counts from the intermetallic:

$$\beta_I = (28 \pm 4) \text{ Bq} \quad (5.45)$$

From this value we can calculate the specific activity of the intermetallic:

$$\beta_{S,I} = (0.93 \pm 0.14) \text{ Bq/mg} \quad (5.46)$$

In order to check if our considerations are correct we can see if, with the specific activities for tin and intermetallic, we are able to reproduce the activity in the whole bulk.

For the intermetallic the activity is:

$$\beta_{T,I} = (62 \pm 9) \times 10 \text{ Bq} \quad (5.47)$$
For the tin the activity is:

$$\beta_{T,T} = (39 \pm 4) \times 10^9 Bq$$  \hspace{1cm} (5.48)

So the total activity from the bulk is:

$$\beta_{B} = (101 \pm 25) \times 10^9 Bq$$

that is just like what we found looking at the activity of the center.

This fact shows that not the whole amount of holmium is in the intermetallic slides, but it is also spread in the tin.

Figure 5.8: Spectrum showing the presence of $^{166}Ho$ in the crucible and in the powders

Comparing the activity we measured from the residual part of the crucible and from various dust with the activity estimated for the bulk, we can conclude that using this method for reducing and making an absorber we have more than 90% of efficiency in recovering the radioactive isotope.
5.6 Generalization of superconducting alloy preparation

The aim of this step is to prepare the superconducting bulk containing the radioactive source, from which the absorber has to be cut. In order to prevent losses, since the amount of radioactive material is negligible and is completely spread inside the crucible, it is important that all the reaction for the preparation of the alloy would take place in the same crucible of the reduction.

It is of main importance that the radioactive atoms are uniformly dispersed inside the superconducting material, in such a way that each atom has the same surrounding and so the decay response be independent form the location. For this reason the amount of radioactive isotope has to be in the range of the metal solubility inside the material bulk, or it has to be possible this element to substitute another element in the crystal lattice of the superconductor (as it will be described in the following section).

We can have two possibilities or M is a superconductor itself or prepare a superconductor containing M.

In the first case, it is important, before opening the crucible, to recover in the bulk, all the radioactive metal deposited on the inner surfaces, this is possible moving and flipping the ampoule several times in an induction furnace at a temperature higher than the melting point of M. The temperature has to be kept under the formation temperature of every compound of the M-isotope system, otherwise the radioactive source may be concentrated in small intermetallic crystals.

In the second case the M is not a superconductor itself, so we need to find an alloy containing this metal in which the radioactive source may be considered as an impurity, this could be a binary alloy, or a more complex compound. Once chosen the proper alloy, all the elements present are weighted in stoichiometric amount (valuing the residual metal already in the crucible) in order to have a single bulk phase. When everything is ready, the crucible is opened and, as soon as possible to prevent contamination or re-oxidation of the radioactive metal, it is filled with the weighted elements and closed again by arc melting in pure argon atmosphere and then it is heated in an induction furnace at high temperature moving and flipping in order to gather all the radioactive isotope on the inner surface. After this step it is sealed in quartz ampoule and, following the phases diagram of that system, it is set in a furnace at the right temperature and for a suitable time to permit the phase formation. In the end the crucible may be opened and it is possible to work the bulk for the preparation of the absorber.
In the case in which it is important to have a particular superconductor as absorber, due to already defined performance, this method can be adapted. If the chosen superconductor doesn’t contain the reductant M, after the first step this metal can be taken out from the crucible and replaced with the metal/elements to have the suitable superconductor. In fact due to the equilibrium vapor pressure, as explained before, at a fixed temperature a fixed number of atoms is in gaseous form and these atoms will deposit as a thin layer when the temperature decreases. During the second step the material for the superconductor interacts with the film deposited on the inner surface, this film is composed mainly by the radioactive atoms and by the reductant atoms, obviously the quantity of this elements has to be estimated and kept below the maximum solubility in the superconducting materials in order to treat them as impurity. This technique will give a lower efficiency compared to the case in which the superconductor is/contains the reductant M, but it is much more adaptable, without any restriction on the choice of the final material. The efficiency will depend on the temperature reached during the first step, since from that the pressure due to the reduced material depends.

5.7 Choice of suitable superconductor

In the last chapter I showed the possibility to obtain a superconductor absorber containing $^{163}\text{Ho}$ atoms widely spread inside using yttrium as reductant and career. The second step is to analyze the features of superconductors containing yttrium in order to identify the best one. The preparation needs to be done in closed Tantalum crucible for keeping the radioactive source inside, so it is important that the elements melted would not attach the Tantalum, above all for the purity of the sample. The transition temperature has to be higher than 100 mK, this comes from the necessity to have a superconducting absorber at the work temperature of the sensor used (TES sensor working around 100 mK). The superconducting alloy has to be a chemically and structurally “stable”, in other words the sample has to be easy to work without damage it, hard, poorly reactive with oxygen in the air and the phase must not decompose in other phases. Another requirement that comes from the rule as photons detector is the stopping power, it has to ensure the 99% efficiency for 3 KeV X-rays in the smaller range as possible, this leads to use element with high Z value and alloys with high density. In this chapter I present the work done to find the best absorber and describe
the features of the superconductors tested.

5.8 \(Y_3Rh_4Sn_{13}\)

This superconductor we decided to use belongs to Rhodium Stannides [91], [92] whose family has several phases featuring interesting properties besides superconductivity, such as ferromagnetism and reentrant superconductivity.

Elements used in this investigation were the commercially available pure metals; purity of 99.9 wt. for the yttrium, rhodium and tin. Y and Sn, were used in form of turnings, freshly machined under argon.

To avoid any weight losses due to possible volatilization during reaction and melting, the alloy was prepared in sealed Ta crucibles. The metals were pressed together into an outgassed Ta crucible which was closed by arc welding under a flow of pure argon; the Ta crucible was then sealed under vacuum in a quartz ampoule and the alloy heat treated melted in a resistance furnace heating up to about 1200 °C, and, to ensure homogeneity, annealed at 700 °C (in a resistance furnace) for 20 days, then cooled to room temperature.

X-ray examination was performed on powders, using a Guinier-Stoe camera with Cu K radiation; pure silicon was used as internal standard (\(a = 5.4308 \text{ Å}\)). The intensities of X-ray reflections were compared with those calculated by means of the LAZY-PULVERIX program [96].

![Figure 5.9: Superconducting transition of \(Y_3Rh_4Sn_{13}\)](image)
powder pattern was well indexed with the body-centered tetragonal cell Er3Rh4Sn13-type [91] [92]; lattice constant values we found are a = 13.757(1), c = 27.511(2) Å.

Metallographic examination was performed with standard techniques; due to the good stability of these samples (specimens) in moist air, no particular care was necessary in their handling. Further identification of the phases was carried out with the aid of electron microscopy (SEM) and microprobe Micrographic investigations with optical and electron microscopy (SEM), and results from semiquantitative analysis carried out by EDX microprobe. From this sample a small rod was cut and polished for measuring the superconducting transition. In fig. 5.9 the transition is showed, there are two critical temperature, the first, due to the presence of tin in excess, at 3.7 K and the second of the intermetallic compound at 3.1 K.

Figure 5.10: SEM image of $Y_3Rh_4Sn_{13}$
5.9 Low temperature characterization of \(Y_3Rh_4Sn_{13}\) as absorber

Three features are especially important for a good absorber: the low heat capacity, the thermal conductance and the stopping power. In general superconducting absorber are element such as tin or rhenium, superconducting alloy have been not tested until now. The specific heat of Rhodium Stannides has been measured by Willekers et al. [93] for \(Er_xY_{1-x}Rh_{1.1}Sn_{3.6}\) at temperature below 100 mK. For a sample of pure erbium alloy they calculate (subtracting the hyperfine interaction of \(^{167}Er\) nuclei) that the heat capacity is, for temperature below 200 mK, below 1 \(J/mol(Er)K\), but for so low temperature their measurement are not completely reliable.

The x-ray stopping power in first approximation, depends on the material density and on the number of electrons. The Rhodium Stannides are composed by a large quantity of tin, with \(Z=50\) with rhodium (\(Z=45\)) and \(Y\) (\(Z=39\)), the density is 6.86 g/cm\(^3\), therefore this alloy can have a good stopping power. Since there are no calculation about the \(Y_3Rh_4Sn_{13}\) stopping power in order to estimate the suitable dimension of the absorber, the tin stopping power can be considered. In fig. 5.11 therefore considering 6 keV photons to have 99 % stopping power 0.015 mm are necessary.

Since \(Y_3Rh_4Sn_{13}\) is never been used as microcalorimeter absorber it is important to understand how it behaves in thermalizing the energy released. For this reason an absorber of this alloy has been prepared, its volume was 0.6 x 0.5 x 0.05 mm\(^3\), then it was glued to an Ge-NTD sensor [98]. This sensor permits to make an analysis of thermalization efficiency in a wide range of temperature (see appendix B for the Ge-NTD 19 used in these measurement).

These measurements were performed in a dilution refrigerator and the acquisition electronics used for NTD sensors is described in [94]. An \(^{55}Fe\) source was used to irradiate the detector and the analysis has been done on the \(K\alpha\) line at 5.9 keV.

The thermalization efficiency was estimated (no precise measurement are available at the moment) looking at the pulse shape, in particular at the decay time. In fact for a not thermalized energy the pulse has a long tail. The measurements were made down to 20 mK, the effect of not well thermalized pulse didn’t appear, therefore this alloy, in this first approximation measurement, seems to permit experiments down to very low temperature.

For an estimation of absorber heat capacity I acquired several IV curves
from the linear part at low bias voltage, the resistance at the working temperature was calculated to set the R(T) curve (see appendix B). From the same sets of data other curves came out, the RV curve and the PV curve. These data permit to calculate the PT curve, from which it was possible to calculate the thermal conductance to the bath of the detector:

\[ P(T) = \frac{G_0}{\beta + 1} \left( T^{\beta+1} - T_s^{\beta+1} \right) \] (5.50)

From the fit of the PT curves for different temperatures the value of \( G_0 \) has been calculated and for all the data set the obtained values gave the best estimation of \( 4.8 \times 10^{-8} \) W/K\(^{\beta-1} \). In fig. 5.13 several PT curves are shown.

The thermal conductance is useful to calculate one of the most important detector properties, the heat capacity. To have this information several pulses at different temperature were acquired. The best condiction were found at 70 mK and therefore the estimation of the heat capacity has been done at that temperature. The pulse decay time depends on the heat capacity through the relation \( \tau = C/G \) where \( \tau \) is the pulse decay time and \( G \) the thermal conductance to the heat sink; therefore C is:
$C = \tau \times G_0 T^3$.

At 70 mK the pulse height corresponding to the 5.9 keV of the $^{55}Fe$ was 225 mV (after the amplification chain x800x10(Stanford)) and there were two relaxation times: a first fast decay with a time constant of 4.2 ms and a second slower of 14 ms. From the previous estimation of $G_0$ the total detector heat capacity is about $10^{-13}$.

No spectrum has been acquired during these test measurements since their aim was only to establish if the superconducting alloy could be used as energy absorber. The results obtained denote the possibility of preparing an energy absorber for $^{163}Ho$ experiment with this alloy since there was no evidence of low temperature slow thermalization and the estimated heat capacity is in the suitable range to permit the detection of low energy x-ray.
Figure 5.13: PT curve at 50 mK with fit
Chapter 6

Last results on Ir-Au TES

6.1 Introduction

The aim of MANU2 experiment is to reach the sensitivity on neutrino mass of about $1 \, eV/c^2$, since the last measurements show that the the absolute mass has to be investigated below $2 \, eV/c^2$. MANU2 is characterized by the use of TES sensors and by the large detectors number, 300. Each detector will have a rhenium single crystal of about 0.6-0.8 mg with dimensions ranging from $0.3\times0.3\times0.3 \, mm^3$ to $0.5\times0.5\times0.5 \, mm^3$, the total mass required has to be around 200 mg. In one year $10^9$ counts are expected which can lead to about 1 eV of neutrino mass sensitivity if the detectors energy resolution is of $\sim 5 \, eV$, or 2-2.5 eV if the energy resolution is 10 eV.

The first test performed on Al-Ag TES produced in the Genoa laboratories showed that we had the possibility to fabricate good quality sensors which could perform to reach the required sensitivity on neutrino mass [56].

A second possibility investigated is the Ir-Au TES, the first results obtained are presented in the next section. The resolution obtained has been better then the one obtained in previous tests, but the noise and the time stability analysis have not been completed so far.

6.2 IrAu TES test with tin absorber

In the development of MANU2 a very important role is played by the choice of the right sensor. The test made on Ir-Au TES have been very promising. The detector used for test was composed by an Ir-Au TES
and a tin absorber of dimensions 0.43 x 0.43 x 0.025 μm$^3$ cut from a poly-crystalline foil. Several measurements were done to understand and optimize the performance of this detector. The procedure to produce the sensor is described in chapter 3 and the superconducting transition is at 83.2 mK.

![Figure 6.1: One of the IV curve taken for the sensor analysis](image)

The acquired pulses corresponding to 5.9 keV were 2.4 V high with a pre-trigger noise of 1.2 mV RMS, the working temperature was set at 82.35 mK. In these conditions the spectrum showed in fig. 6.3 has been obtained. The energy resolution is of 5.9 keV and the $K_\alpha$ line has been separated from the $K_\beta$.

This is, even if preliminar, the best result obtained during the development of MANU2 thermal sensors. This energy resolution, if confirmed even with a rhenium absorber would permit to lower the sensitivity on neutrino mass down to 1 eV/$c^2$. 

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Figure 6.2: Example of the pulse corresponding the 5.9 keV
Figure 6.3: Final 55-Fe spectrum obtained with uc54
Chapter 7

Au:Er sensor: a new possibility for 187-Re experiment

7.1 Introduction

The very promising results obtained with magnetic calorimeters showed in [32] and in [95] denote how these sensors can perform an important role in particles detection. The major problem in the direct search of neutrino mass with $^{187}Re$ is the large rhenium mass necessary to have the suitable activity. Among the properties these sensors have, it is important to note that the maximum sensitivity is proportional to $C_a^{-1/3}$, therefore this weak absorber heat capacity dependence could be promising for a large mass experiment such as $^{187}Re$ direct neutrino mass measurement.

A first problem which arises, is the fact that magnetism and superconductivity can hardly coexist. Usually a magnetic calorimeter consists of a metallic absorber (typically gold) glued directly over the magnetic sensor. The use of metallic superconducting rhenium as detector absorber is not straightforward for several reasons, both for the sensor good working and both for the increasing of absorber heat capacity. In fact magnetic sensors need to operate inside a magnetic field in order to polarize the paramagnetic atoms, while the same magnetic field can disturb the behavior of the absorber in particular it can drive part of the external zones from the superconducting to the normal state, causing an enhancement of the heat capacity. On the other hand the presence of a superconductor in proximity of the sensor can deform in a dangerous way the field in the sensor itself.

In order to understand if the magnetic sensor could be a possible improve-
ment for the rhenium experiment it is useful to look at the properties of rhenium under magnetic field, this will be described in the next section. After that I will describe two possible solutions I developed with the help of the group of Heidelberg during a period spent there.

### 7.2 Rhenium behavior in magnetic field

The behavior of superconducting rhenium absorber under magnetic field has been investigated by the group of Genoa. The thermalization efficiency $\epsilon$ of a detector at 60 mK was measured in weak magnetic fields. Two different situations were tested, in the first the rhenium has been cooled down below its critical temperature under magnetic field, in the second the field was switched on only when the metal was superconducting.

In the first case, increasing $H$, the efficiency got lower and lower showing that the part of energy rapidly thermalized was diminished by the presence of magnetic field. In fig. 7.1 the efficiency is reported as a function of $H$ and also the specific heat capacity $C(H)/C(0)$, calculated as the ratio between the height of the pulses with and without field, corrected by the corresponding efficiency. On the other hand if the rhenium is already superconducting when the magnetic field is switched on, then up to 20 gauss and for temperatures around 100 mK, there are no deviations in the shape and height of the pulses.

Another question to deal with is the energy thermalization inside the absorber which limits the possible working temperature. As described in chapter 5 the rhenium working temperature can not be below 90 mK. In order to work at lower temperature we decide to try to cover the side of the absorber to be glued to the sensor with 100 nm of gold, this metal layer would thermalize faster the phonons by means of electrons scattering.

The two design are studied in order to test the detector both with the gold layer and without.

### 7.3 Detector design

Two different set-ups have been studied, the first is a modification of the actual magnetic detector design, while the second is characterized by a new configuration for magnetic sensors which is being developed by the group of Heidelberg. In these subsections I will describe the two set ups. The studies for set up design were mainly addressed to minimize the
magnetic field in the region of the absorber and minimize the magnetic field distortions inside the sensor. This investigation was made by means of finite element simulation with the free software FEMM.

### 7.3.1 I set-up

In the normal configuration the magnetic calorimeter is composed by a metallic absorber directly set over the Au:Er sensor; the magnetic field for polarize the paramagnetic atoms is about 3 mT. The case of superconducting rhenium absorber can not be treated in the same way, as it was already described. Therefore I looked for possible configuration in which superconducting absorber and magnetic field can coexist. A first simple solution would be to separate sensor and absorber by a suitable distance. I begun to consider the possibility for a vertical separation between the sensor and the absorber which would permit to minimize the undesired effects.

FEMM simulations were performed on the geometry of the SQUID chip.
Figure 7.2: Picture of the SQUID to be used for Rhenium experiment. To note the presence of field coil directly on the same chip (IPHT-Jena)

showed in fig. 7.2, an important improvement achieved with this configuration, is to have the field coil close to the sensor and therefore limiting the region with magnetic field.

In fig. 7.3 are showed the FEMM simulations with the absorber 20 μm above the sensor. The magnetic field is generated by the current flowing through the coil evaporated on the same chip of the SQUID; the current has been changed to see the following modifications of magnetic field. Fig. 7.4 shows the value of magnetic field in presence of the superconducting absorber and in particular the variation of magnetic field along the lower part of the sensor is showed. The same calculation were performed without the superconductor above the sensor. The comparison of the magnetic field in the two case leads to two main considerations. The magnetic field inside the sensor is disturbed in a weak way by the presence of the superconductor and the magnetic field in the region of the rhenium can be mantained below the 20 mgauss. This would give the possibility to use superconducting rhenium as absorber, but how may the distance between the two detector components be filled? The solution we found is to insert a gold cylinder with 25 μm
diameter and 20 μm height as shown in fig. 7.4 If we consider an rhenium absorber of 3 mg working around 50 mK the contribution to the heat capacity can be written as:

\[ C_{\text{abs-lat}} = 5.4 \times 10^{-14} \text{ J/K} \]  \hspace{1cm} (7.1)
\[ C_{\text{gold-S}} = 1 \times 10^{-13} \text{ J/K} \]  \hspace{1cm} (7.2)
\[ C_{\text{gold-SF}} = 2 \times 10^{-13} \text{ J/K} \]  \hspace{1cm} (7.3)
\[ C_{\text{Au:Er}} \approx 10^{-12} \text{ J/K} \]  \hspace{1cm} (7.4)

The thermal conductance of the gold spacer is estimated as:

\[ G_{\text{gold-S}} \approx 10^{-4} \text{ W/K} \]  \hspace{1cm} (7.5)

These numbers give a very fast rise time of about 10^{-8} s, the decay time depends on the thermal link between the sensor and the bath and can be estimated around 1-10 ms. It is important to consider that small external part of rhenium may drive to normal with an enhancement of heat capacity. The thin gold film would provide an higher thermal conductiv-
Figure 7.4: FEMM simulation for I set-ut

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Figure 7.5: Lowering the magnetic field outside the sensor by means of meander structure

ity from the absorber to the sensor. Therefore we could investigate the efficiency of this film at temperature lower than 90 mK comparing the pulse shapes acquired with and without the film.

7.3.2 II set-up

The set up described in the previous section is not the best configuration for optimizing the magnetic field pattern, since we have reached a compromise in order to have a low magnetic field in the absorber region and a high and homogeneous enough magnetic field inside the sensor. The Heidelberg group is developing a new technique to confine the magnetic field just around the sensor and not in the close regions. This technique has been developed looking at arrays of magnetic calorimeters, in fact the cross-talk problem between two close sensors due to the flux variation in one of them following the absorption of an x-ray can be solved confining the field in a small region around the sensor by means of meanders structure as shown in fig. 7.5

The same configuration may be applied for the rhenium detector. There could be two different possibilities, the first can have the absorber positioned over the Au:Er film with a spacer between (thinner than the one used in the previous set-up. The other needs several steps of evaporation over the rhenium itself as shown in fig. 7.6. The advantages in using this configuration come from the better localization of the field in the sensor.
region and from the reduced amount of gold that can be used. More refined simulation are useful to define better the right detector dimensions.

7.4 Final considerations

The prospective of using magnetic sensor for $^{187}$Re experiment is very attractive, not only for the energy resolution they can obtain, but above all for the possibility to use bigger absorber and therefore reducing the number of channels to reach the desired count rate. Beside that experimental test are to be done to ensure the good working of magnetic sensor and superconducting absorber. From theoretical simulation and from previous experiences on rhenium in magnetic field it looks promising the studied configurations. Other important consequence is the possibility to investigate thermal processes in the rhenium and above all if a metallic gold layer can help in thermalization of quasiparticles. Next future test will give us the answer if Au:Er sensors are a real improvement for $^{187}$Re experiment.
Conclusion

In this thesis different aspects of low temperature detectors have been treated, especially I worked on the improvement of the Transition Edge thermal sensors (TES) and on the preparation of inner source microcalorimeter energy absorber.

Different possibility have been studied for TES superconducting film, beginning with the Ag-Al multilayers with which promising results have already been obtained, Ir-Au and finally Al:Mn and Zn:Mn dilute alloy films. In particular the Ag-Al system has been deeply investigated in order to understand the chemical and physical behavior, this lead to develop a solution for the stability of Ag-Al TES.

The possibility to improve the energy resolution with which radioactive isotope spectra may be acquired can be attained by means of low temperature microcalorimeter with the source directly inside the absorber. A method for inner source absorber preparation has been studied and tested; the efficiency, especially in rare earths recovering on very small amount of initial material (less than nanograms), has been higher than 90%.

Very interesting result in term of IrAu TES microcalorimeter has been presented and the energy resolution of 5.9 eV set an important step towards the required neutrino mass sensitivity.

At the end a new possibility for future $^{187}$Re experiment has been presented. In collaboration with the Low Temperature Detectors group of Heidelberg, the preliminar study to use Au:Er magnetic sensors with superconducting rhenium absorber has been performed and the presented solutions seem quite attractive.
Appendix A

Proximity effect

A.1 Introduction

If a normal metal N is deposited on top of a superconducting metal S and if the electric contact is good, Cooper pairs can leak from S to N therefore transferring superconducting properties to the normal metal too. This proximity effect is actually long range, in fact the thickness $K^{-1}$ of the leakage region is typically $10^3 \text{Å}$.

Several models describing the changes of parameters such as the transition temperature of a proximity bilayer or the spatial variation of the energy gap have been developed. In general for the application of these models in thin film it is important to keep in mind some requirements for sample preparation:

1) the electric contact between N and S must be very good to permit electron pairs migration. Problems arise when an oxide layer is between N and S.

2) the atoms of N must not migrate to S and vice versa (especially for two reasons: a small concentration of N in S lower the S’ transition temperature and the electron transmission properties at the boundary can be affected)

3) The mean free paths of both $l_S$ and $l_N$ are very important.

A complete review of proximity effect is found in Parks [97] by Deutscher and de Gennes. In this appendix I will follow the scheme proposed there.
A.2 Transition temperature of an N-S bilayer in zero magnetic field

A.2.1 Thick S layers: definition of extrapolation length $b$

The transition temperature of N-S bilayer with an S layer od thickness $d_S$ much larger than the coherence length in S at T=0 K, $\xi_0 S$ is close to the unperturbed value $T_{cS}$ and therefore the Ginzburg Landau equation can be applied to the S side.

At the transition the order parameter $\psi$ is small and we can use the linearized equation for $\psi$:

$$-\alpha(T_{cNS})\psi = \frac{\hbar^2}{2m} \frac{d^2\psi}{dx^2} \quad (A.1)$$

valid on the S side, for $0 \leq x \leq d_S$ and $\alpha$ is proportional to the shift in the transition temperature:

$$\alpha(T_{cNS}) = (T_{cNS} - T_{cS})\alpha' \quad (A.2)$$

In the vicinity of the metal, equation (8.1) breaks down, but the effects of the proximity to a superconductor can be included in the boundary conditions:

$$\left(\frac{d\psi}{dx}\right)_{x=0} = \frac{\psi}{b} \quad (A.3)$$

$$\left(\frac{d\psi}{dx}\right)_{x=d_S} = 0 \quad (A.4)$$

The extrapolation length $b$ depends on the thickness and other properties of N layer, but not on the thickness of S layer (for large $d_S$). The solution of $\psi$ in S is:

$$\psi = \text{const} \cos[q(x - d_S)] \quad (A.5)$$

together with the conditions:

$$\frac{\hbar^2}{2m} q^2 = -\alpha = \alpha'(T_{cS} - T_{cNS}) \quad (A.6)$$

$$qtan(qd_S) = \frac{1}{b} \quad (A.7)$$

For large $d_S$ equation (8.6) becomes:

$$q = \frac{\pi}{2}(d_S + b) \quad (A.8)$$
and therefore the transition temperature of the bilayer is:

\[ T_{cNS} = T_{cS} - \frac{\pi^2 h^2}{8 m \alpha'} \frac{1}{(d_S + b)} \]  

(A.9)

### A.2.2 Microscopic calculations

We assume that the transition temperature \( T_{cNS} \) of the NS bilayer corresponds to a second order phase change. Then for \( T = T_{cNS} \) the Cooper pair amplitudes \( F(r) = \psi_1^*(r) \psi_1(r) \) are small everywhere and are ruled by a self-consistent equation. In each metal we make an approximation of the BCS type and therefore we are writing an electron-electron interaction, described by a coupling constant \( V(r) \):

\[ V(r) = V_S \quad (x > 0; \ S \ side) \]  

(A.10)

\[ V(r) = V_N \quad (x < 0; \ N \ side) \]  

(A.11)

Since S is a superconductor the interaction between electrons is positive \( (V_S > 0) \) while in the normal metal the sign for \( V_N \) can be of either sign. The interaction is cut off at a frequency \( \omega_D(x) = \omega_{DS} \) or \( \omega_{DN} \). Then the linearized equation for the pair potential \( \Delta(x) = V(x)F(x) \) has the form:

\[ \Delta(r) = V(r)k_B T \sum_\omega \int G_\omega(rr')G_{-\omega}(rr')\Delta(r')dr' \]  

(A.12)

where \( G_\omega \) is a one electron thermal Green’s function in the normal phase and \( h\omega = (2n + 1)\pi k_B T \). The maximum temperature at which there is a non zero \( \Delta(x) \) is the transition temperature \( T_{cNS} \). In one dimensional geometry, the previous equation may be written:

\[ \Delta(r) = V(r)k_B T \sum_\omega \int H_\omega(xx')\Delta(x')dx' \]  

(A.13)

where \( x \) is the direction normal to the layer and \( H_\omega(xx') \) is the hamiltonian of the system. The boundary conditions at the interface:

\[ \frac{\Delta}{N_N} = \frac{F}{N} \quad continuous \]  

(A.14)

\[ \frac{D}{dx} \frac{dF}{dx} \quad continuous \]  

(A.15)

where \( N , N_S \) and \( N_N \), are the densities of states at the Fermi level in the normal phase. While at the outer surfaces the boundary condition are:

\[ \frac{\Delta}{N_N} = 0 \]  

(A.16)
If the metal N is clean, i.e. its mean free path is large compared to the coherence length $\xi_N$, the probability amplitude $F = \psi_\uparrow \psi_\downarrow$ for finding a Cooper pair at a distance $-x$ from the NS boundary has the asymptotic form:

$$F = \phi(x) e^{-K|x|} \text{ for } |x| \text{ large}$$  \hspace{1cm} (A.18)

where $\phi$ is a slowly varying function of $x$ and:

$$K^{-1} = \frac{\hbar v_N}{2\pi k_B T}$$  \hspace{1cm} (A.19)

where $v_N$ is the Fermi velocity in N and T the temperature.

The case in which N is "dirty" ($l_N < \xi_N$) is more interesting since it approaches better the experimental situation, and therefore the experimental uncertainties on the interface properties are minimized. In this limit the leakage of pairs is controlled by a diffusion process, therefore a diffusion coefficient is introduced, $D = \frac{1}{3} v_N l_N$. The asymptotic form for the probably amplitude is the same of the clean limit, but in this case the coherence length has the form:

$$\xi_N = \left( \frac{\hbar D}{2\pi k_B T} \right)^{1/2} = \left( \frac{\hbar v_N l_N}{6\pi k_B T} \right)^{1/2}$$  \hspace{1cm} (A.20)

In the case in which $V_N$ is 0, the pair potential $\Delta(r)$ vanishes in N, but the condensation amplitude $F(r) = \Delta(r)/V(r)$ has a finite tail in the normal region. $F(r)$ is given by the integration of Eq. (8.12) over the S region only.

In the case of finite $V_N$ and for $T = T_{cNS} > T_{cN}$, the form of $F$ in N is again exponential and the thickness $K^{-1}$ is the smallest root of:

$$\log\left( \frac{T}{T_{cN}} \right) = \sum_{n\geq0} \left[ -\frac{1}{n + \frac{1}{2}} + \frac{1}{n + \frac{1}{2} - K^2 \xi_N^2} \right]$$  \hspace{1cm} (A.21)

These results are valid only when the linearized equation for $\Delta$ is applicable, in particular $\Delta < k_B T$. This condition is satisfied if $T = T_{cNS}$, since at the transition $\Delta$ is everywhere infinitesimally small. In many cases it is also satisfied at temperature smaller then $T_{cNS}$ because $\Delta$ is small on the N side, but for $T << T_{cNS}$ a non linear equation should be used instead of Eq. (8.12).
A.2.3 \( T_{cNS} \) calculation with Cooper limit

If both the normal metal layer and the superconducting layer are thin, \( d_S < \xi_S \) and \( d_N < \xi_S \), and therefore it is possible to consider \( \Delta_S(x) \) and \( \Delta_N(x) \) constant inside the two films.

A simple argument to calculate \( T_{cNS} \) is to consider that the time one electron spends inside N and inside S is respectively proportional to \( d_S \) and \( d_N \), and the BCS effective interaction parameter \( (NV)_{eff} \) is the sum of the parameters \( (NV)_S \) and \( (NV)_N \) weighted by \( d_S \) and \( d_N \). Thus the average coupling constant can be written as:

\[
(NV)_{eff} = \frac{N_N V_N d_N + N_S V_S d_S}{N_N d_N + N_S d_S} \tag{A.22}
\]

and it can be used in the BCS formula.

In the case in which the coupling constant inside the metal is 0, the effective coupling constant becomes:

\[
(NV)_{eff} = \frac{N_S V_S d_S}{N_N d_N + N_S d_S} \tag{A.23}
\]

and thus the transition temperature is given by:

\[
T_C = 1.14 \Theta_D e^{-\frac{1}{[N(0) V]_{eff}}} \tag{A.24}
\]

A.2.4 \( T_{cNS} \) calculation with de Gennes-Werthamer limit

The de Gennes-Werthamer limit is used in the case of thick films, that is when \( d_S > \xi_S \) and \( d_N > \xi_N \). The self-consistent equation for \( \Delta(x) \) is:

\[
\Delta(x) = V(r)k_B T \sum_\omega \int_0^{d_S} H_\omega(xx')\Delta_S(x')dx' + \int_{-d_N}^0 H_\omega(xx')\Delta_N(x')dx' \tag{A.25}
\]

In order to solve this equation we need to make two hypothesis:
- \( \Delta(x) \) is the same solution as for semiinfinite films -\( \Delta(x) \) has sinusoidal form in S and exponential form in N (as suggested by Ginzburg-Landau equations)

Thus we can rewrite \( \Delta(x) \) as:

\[
\Delta_S(x) \propto \cos \frac{d_S - x}{\xi_S(T)} \quad x > 0 \tag{A.26}
\]

\[
\Delta_S(x) \propto \sinh \frac{d_N + x}{\xi_N(T)} \quad x < 0 \tag{A.27}
\]
where $\xi_S(T)$ and $\xi_N(T)$ are the solution of:

\[
\ln \frac{T_{cS}}{T} = \psi\left(\frac{1}{2} + \frac{\hbar D_S}{4\pi kT\xi_S^2}\right) - \psi\left(\frac{1}{2}\right) \quad (A.28)
\]

\[
\ln \frac{T_{cN}}{T} = \psi\left(\frac{1}{2} + \frac{\hbar D_N}{4\pi kT\xi_N^2}\right) - \psi\left(\frac{1}{2}\right) \quad (A.29)
\]

Here $\psi$ indicate the digamma function.

The boundary condition at the interface has the following form:

\[
\frac{1}{\xi_S(T)} \frac{d\xi_S}{dT} = \frac{1}{\gamma} \tanh \frac{d\xi_N}{dN} \quad (A.30)
\]

where

\[
\gamma = \frac{N_SD_S}{N_ND_N} = \frac{\sigma_S}{\sigma_N} \quad (A.31)
\]

$\sigma$ is the normal state conductivity. From this relation it is possible to calculate $T_{cNS}$ since $\xi_S$ and $x_{iN}$ are temperature dependent. In principle the de Gennes-Whertarmer equation has no free parameters. In practise, however an adjustment factor has to be included to explain the experimental results.

### A.2.5 $T_{cNS}$ calculation with Usadel model

The Usadel theory is a microscopic theory and it is based on the assumption that electrons travel diffusively through the metal. The electron states are described through an energy variable $E$ and the superconducting state is described by the function $\Theta(x, E)$, where $x$ is the position coordinate. The variable $\Theta$ is complex and ranges from 0 for the normal state, to $\pi/2$.

The Usadel equation for $\Theta$ is:

\[
\frac{\hbar D_S}{2} \frac{\delta^2 D_S}{\delta^2 x} + iE\sin\Theta - \left[\frac{\hbar}{\tau_{sf}} \left(\frac{\delta\phi}{\delta x} + \frac{2e}{\hbar} A_x\right)^2\right] \cos\Theta \sin\Theta + \Delta(x) \cos\Theta = 0 \quad (A.32)
\]

with

\[
\Delta(x) = N_S V_{eff} \int_0^{\hbar \omega_D} dE \tanh \frac{E}{2k_BT} \text{Im}[\sin\Theta] \quad (A.33)
\]

where $\tau_{sf}$ is the spin-flip time, $\phi$ is the superconducting phase, $\hbar \omega_D$ is the Debye energy and $A_x$ is the vector potential.

A simple, but interesting case is that of a uniform superconductor where there is no spatial dependence and no pair breaking from magnetic impurities, field or current. In this case the first and third term of Eq. (8.32)
are zero and $\Theta$ corresponds to $\Theta_{BCS}(E) = |E|/(E^2 - \Delta^2)^{1/2}$. With these simplification the equation for $\Theta$ can be linearized:

$$\frac{\hbar D_S}{2} \frac{\delta^2 D_S}{\delta^2 x} + iE\Theta + \Delta = 0 \quad (A.34)$$

At the interface the spectral current has to be conserved, this means:

$$\sigma_N \frac{\delta \Theta_N}{\delta x} = \sigma_S \frac{\delta \Theta_S}{\delta x} = \frac{G_{int}}{A} \sin(\Theta_N - \Theta_S) \quad (A.35)$$

$G_{int}/A$ is the electrical conductance per unit area. In this scheme the transition temperature of a bilayer can be calculated from:

$$\frac{T_{cNS}}{T_{cS}} = \left[ \left( \frac{k_B T_{cS}}{1.13 \hbar \omega_D} \right)^2 + \left( \frac{k_B T_{cS}}{1.13 \tau} \right)^2 \right] d_{NN}/2 d_{NS} \quad (A.36)$$

where

$$\tau = \frac{G_{int}/A}{4\pi G_K} \left( \frac{1}{d_{NN}N_N} + \frac{1}{d_{NS}N_S} \right) \quad (A.37)$$

with $G_K$ as the quantum conductance.

A.2.6 $T_{cNS}$ calculation with McMillan model

This model considers a potential barrier between the S and N metals. It has one free parameter $\sigma$ which describes the transmission of electrons across the barrier. The metals are supposed to be thin so that the superconducting properties don’t change across the metals. In such case the transition temperature $T_{cNS}$ is given by:

$$T_{cNS} = T_{cS} \left( \frac{k_B T_{cS} \pi}{2 \gamma (\Gamma_N + \Gamma_S)} \right)^{\Gamma_S/\Gamma_N} \quad (A.38)$$

with $\gamma = 1.781$ and

$$\Gamma_{S,N} = \frac{\hbar \nu_{S,N}}{2} \left( \frac{\sigma}{B(\frac{l}{d_{S,N}})} \right)^{1/d_{S,N}} \quad (A.39)$$

$B$ is a dimensionless function of the ratio of the mean free path $l$ to the film thickness $d$. This relation is valid in the Anderson limit $\Gamma_S + \Gamma_N >> k_B T_{cS}$. 

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Appendix B

Ge-NTD sensors calibration

B.1 I-V curves acquisition

Neutron Transmutation Doped (NTD) germanium sensors are commonly used as thermistors for low temperature detectors in several experiments such as radiation detection and astrophysical measurements. These sensors are prepared irradiating natural germanium samples with thermal neutrons. They are used as thermal sensors since they have a well-known dependence of resistivity on temperature, which for low temperature the transport mechanism is the hopping. The predicted relation between temperature and resistivity is in the hopping regime for doped Ge:

\[ \rho = \rho_0 \exp \left( \frac{T_0}{T} \right)^{1/2} \]  

(B.1)

where \( \rho_0 \) and \( T_0 \) are parameters which depend on the ratio of acceptor and donor concentrations.

During the test on the superconductor for the \(^{163}Ho\) experiment, the sensor used was a Ge-NTD. To calibrate that several I-V curves have been acquired at different temperatures.

The first part of these curves shows a linear dependence between resistance and temperature, from these data a linear has been done and the resistance at that temperature was calculated. In fig. B.1 all the curves acquired during one of the runs are showed and the typical behavior of NTD sensor appears.
Figure B.1: I-V curves acquired for a Ge-NTD sensor; from the low current the curve is linear and from those point the sensor resistance has been calculated.

## B.2 Thermometer calibration

The feature of Ge-NTD sensor are described in [98], the dopant concentration is decided in order to have the best performances in different temperature ranges. The sensor used in these measurement was Ge-NTD12.

From the fit presented in fig. B.2 the parameter found are:

\[
R_0 = \rho_0 \times \frac{l}{A} = (0.44 \pm 0.17) \times 10^3 \, \Omega
\]

\[
T_0 = (8.1 \pm 0.5) \, K
\]

where \( R_0 \) takes into account the geometrical shape of the sensor (length \( l = 250 \, \mu m \) section \( A = 50 \times 50 \, \mu m^2 \)). The parameter \( T_0 \) corresponds to the value predicted from the theory \([?]\) of 7.84 K for NTD-12, while the \( \rho_0 \) , which is supposed to be 0.02 \( \Omega \) cm, is one order of magnitude bigger as calculated with the previous geometrical parameters (in fact \( R_0 \) is supposed to be \( 0.02 \times 10^3 = 20 \Omega \)). The reason for that may derive from
Figure B.2: Ge-NTD calibration curve

geometrical defects which reduce the section A of conduction in a region inside the detector.
Therefore the relation between the temperature and the resistance of the sensor used is:

$$R = 5 \times 10^2 \exp \left( \frac{8.1}{T} \right)^{1/2}$$  \hspace{1cm} (B.2)

from which the equation to calculate the temperature for a known resistance:

$$T = \frac{8.1}{\left( \ln \frac{R}{5 \times 10^2} \right)^2}$$  \hspace{1cm} (B.3)
Acknowledgements

A lot of people have contributed at this thesis both from the professional and psychological point of view. In the first group there are persons which helped me in improving my knowledge in different field. I will thanks all of them in alphabetic order: Emilio Bellingeri and Daniele Marre’ for PLD of AgAl alloy thin film, Andrea Chincarini for all the time he spent making XPS analysis, Riccardo Ferrando who showed me the secrets of Molecular Dynamics simulations, Gaetano Gallinaro (a second father) for all the test we made on new possible TES, Pietro Manfrinetti who gave me an indespensable help for the alloy preparation and above all because he believed in me during the holmium reduction processes (even when everything seemed wrong), Chiara Tarantini for the x-ray analysis and finally all the people in the Heidelberg group with which I worked in a pleasant and professional way.

Now I want to thank all of my friends and coworkers, beginning from the people working in L103: Adriano, Chiara, Daniele, Davide, Luigi, Pietro, Riccardo, Simeone and Lilli.
All my evergreen friends with who I could complain everytime something wrong happened in the lab Amanda, Andrea, Anna and Cristina and my PhD student collegues. Then all the friends which could not understand why I had to work also on Saturday and all my train friends.
Thank to Marco for his help and to my friend Hsin-Yi always present even if on the other side of the ocean.
Finally I want to thank my family.
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