Implantation and Ion Beam Analysis of thin Gold films in the framework of the MARE experiment

Alexandra Furtado Guerreiro

Dissertação
Mestrado Integrado em Engenharia-Física
2014
Implantation and Ion Beam Analysis of thin Gold films in the framework of the MARE experiment

Alexandra Furtado Guerreiro

Dissertação
Mestrado Integrado em Engenharia-Física
2014

Orientador: Prof. Dr. Maria José Ribeiro Gomes
Acknowledgements

I would like to thank Centro de Física Nuclear da Universidade de Lisboa as my host institution, Campus Tecnológico e Nuclear - IST for allowing me the use of the experimental research facilities, and Istituto Nazionale di Fisica Nucleare/Genoa also for allowing me the use of the experimental research facilities and producing the samples.

I wish to express my sincere thanks to Dr. Eduardo Alves, Dr. Nuno Barradas, Jorge Rocha, Dr. Luís Alves, Dr. Rui Coelho, Dr. Norberto Catarino, Dr. Flavio Gatti, Dr. Michele Biasotti, Dr. Giulio Pizzigoni and Dr. Dario Corsini for their work, support and suggestions.

A special thanks to my supervisor Dr. Maria José Ribeiro Gomes for her guidance, persistent help and advices.

None of this work could have been done without the support of my friends and family, and so I dedicate this thesis to them.
Resumo

A experiência Microcalorimeter Arrays for a Rhenium Experiment (MARE) tem como objetivo a medição da massa do neutrino do electrão com uma sensibilidade de 0.1 eV e ser uma alternativa competitiva à experiência KATRIN, que utiliza como detector um espectrômetro. MARE usará a técnica de calorimetria, onde os absorbedores dos microcalorímetros serão constituídos por um isótopo com uma baixa energia de transição, Q, no decaimento beta. Esta experiência irá usar o decaimento beta do isótopo $^{103}$Re, devido ao seu valor baixo de Q, à sua abundância isotópica e reduzida capacidade térmica [1]. O potencial deste isótopo para uma experiência calorimétrica na medição da massa do neutrino já foi demonstrada pelas experiências MANU [2, 3] e MIBETA [4,5].

Alternativamente, HOLmium EC MEaSurement for electron neutrino mass measurement (HOLMES) foi desenvolvida pela colaboração MARE, onde a determinação directa da massa do neutrino será conseguida usando o processo de captura de electrões do isótopo $^{163}$Ho, devido ao seu baixo valor de Q e à sua pequena vida-média, utilizando, também, microcalorímetros criogénicos.

O grande avanço da tecnologia microcalorimétrica e as suas aplicações devem-se ao desenvolvimento dos microcalorímetros TES como espectrômetros de imagem. Hoje em dia, a tecnologia com microcalorímetros criogénicos pode ser usada numa experiência de larga escala com excelente resolução de energia e de tempo [6]. Os microcalorímetros de Sensor de Borda de Transição (TES) são a escolha para o detector numa experiência com o objetivo de medir o decaimento radioativo de captura de electrões do hólio, fornecendo o valor da massa do neutrino do electrão. Uma fonte de $^{163}$Ho incorporada num microcalorímetro TES para medição de massa do neutrino está em curso no âmbito da colaboração das experiências MARE e HOLMES.

Para optimizar os parâmetros do detector de HOLMES, é importante conhecer as alterações das propriedades térmicas do absorbedor quando implantado com $^{163}$Ho. Uma vez que o $^{163}$Ho é produto da activação de neutrões do $^{162}$Er, também é necessário conhecer os efeitos de érbio, quando incorporado no absorbedor. Particularmente, a propriedade de interesse é a capacidade térmica do Uma opção que está a ser investigada é a implantação do isótopo radioativo num de ouro, dado que o ouro tem sido utilizado com sucesso em outras aplicações [6], e as suas características correspondem aos requisitos necessários para uma experiência como HOLMES.

Este trabalho, realizado no âmbito das actividades de I&D do projeto PTDC/FIS/116719/2010, consistiu na implantação de íons metálicos não-radioativos de Ho e Er em filmes finos de ouro e na sua caracterização usando Análises de Feixe de íons e a Análise de Difração de raio-X, e também em medições de baixa temperatura, utilizando um refrigerador de diluição equipado com um SQUID para medir a região de transição do material superconductor de um dispositivo TES.

Para uma elevada estatística na região próxima do valor Q é importante um elevado número de decaimentos, o qual implica uma concentração elevada de $^{163}$Ho e de $^{162}$Er. A motivação deste trabalho consistiu em aumentar, uma ordem de grandeza, a concentração de ambos os isótopos, Ho e Er, em relação aos valores usados numa experiência anterior [7]. Desta forma, é possível estudar os fenómenos inerentes ao processo de implantação e, ao se testar doses cada vez mais elevadas, de acordo com os requisitos de HOLMES, é possível optimizar o processo de implantação. Os parâmetros originalmente estabelecidos para implantação foram plenamente alcançados na experiência anterior, em 12 amostras, sem danos da amostra ou contaminação.

Os resultados deste trabalho mostraram que não foi possível atingir os parâmetros de implantação conforme planeado e garantir uma elevada qualidade dos filmes. Além disso, os parâmetros de implantação foram fundamentais para no futuro contornar-se processos de sputtering na superfície do filme durante o processo de implantação e a degradação física e química das propriedades do filme.

Palavras-chave: massa do neutrino, microcalorímetro, TES, filmes finos
Abstract

The Microcalorimeter Arrays for a Rhenium Experiment (MARE) aims at providing a competitive alternative to the spectrometric approach, used by KATRIN experiment, in the effort of measuring the neutrino mass with a sensitivity as low as 0.1 eV. MARE will use the calorimetric technique, where the thermal microcalorimeters absorbers will contain a low transition energy $Q$ beta decaying isotope. This experiment will use $^{187}$Re $\beta$-decay, due to its low $Q$ value, isotopic abundance and reduced thermal capacity [1]. The potential of this isotope for a calorimetric neutrino mass experiment has already been demonstrated by MANU [2, 3] and MIBETA [4,5] experiments.

A parallel experiment is being developed by the MARE collaboration, called HOLmium EC MEasurement for electron neutrino mass measurement (HOLMES) experiment, where the electron neutrino direct mass determination will be achieved using the electron capture process of $^{163}$Ho, due to its low $Q$ value and its short half-life, also using cryogenic microcalorimeters.

There has been huge improvement in microcalorimetric technology and its applications due to the development of TES microcalorimeters as imaging spectrometers. Nowadays, cryogenic microcalorimeter technology can be used in a large scale experiment with excellent energy and time resolutions [6]. Transition Edge Sensor (TES) microcalorimeters are the detector choice for an experiment with the goal of measuring the electron capture decay of holmium radioactive, providing the value of electron neutrino mass. A source of $^{163}$Ho embedded in a TES microcalorimeter for neutrino mass measurement is under course on the framework of the MARE collaboration and HOLMES experiment.

To optimize the detector parameters for HOLMES it is important to know the changes in the absorber thermal properties when $^{163}$Ho is embedded in the detector. Since, $^{163}$Ho is a product of the neutron activation of $^{162}$Er it is also necessary to know the effects of erbium when embedded in the absorber. The particular property of interest is the absorber heat capacity. One option that is being investigated is to implant the radioactive isotope into a gold absorber, as gold has been successfully used in other applications [6], and its characteristics match the requirements needed for an experiment such as HOLMES.

This work, performed within the R&D activities of the project PTDC/FIS/116719/2010, consisted on the ion implantation of metallic non-radioactive Ho and Er in gold thin films and its characterization using Ion Beam Analysis and X-Ray diffraction analysis, and also low temperature measurements using a dilution refrigerator equipped with a SQUID to measure the transition region of a TES device.

In order to have high statistics at the $Q$ value region it is important to have a high number of decays, which implies a high concentration of both $^{163}$Ho and $^{162}$Er. The motivation of this work was to increase the concentration of both Ho and Er, one order of magnitude higher than the ones used in a previous experiment [7]. In this way, it is possible to study the inherent phenomena to the implantation process and, when testing increasingly high doses, according to HOLMES requirements, it is possible to optimize the implantation process. The parameters originally set for implantation have been fully achieved in this previous experiment in 12 samples, with no sample damage nor sputtering nor contamination processes.

The results of this work showed that it was not possible to achieve the implantation parameters as planned guaranteeing the high quality of the thin films. Furthermore, implantation parameters were a paramount in order to avoid sputtering processes on the film surface during ion implantation and further physical and chemical degradation of the film properties.

Keywords: neutrino mass, microcalorimeter, TES, thin films
Table of Contents

1. Brief Introduction ................................................................................................................. page 1
2. Neutrino Physics .................................................................................................................. page 3
  2.1. Beta Decay ....................................................................................................................... page 5
    2.1.1. Density of States ........................................................................................................ page 8
    2.1.2. Decay Rate ................................................................................................................ page 11
    2.1.3. Energy Spectrum of Electron Capture Decay ....................................................... page 14
3. Microcalorimeters with Transition Edge Sensors ............................................................... page 17
  3.1. Microcalorimeter Working Principle ............................................................................. page 17
  3.2. Microcalorimeter Modelling ......................................................................................... page 20
  3.3. Transition Edge Sensor Detector .................................................................................. page 25
4. Microcalorimetric Measurements of the β-decay for neutrino mass direct determination .. page 30
  4.1. β-DECAY OF 187RE ISOTOPE ....................................................................................... page 31
  4.2. The MARE project ....................................................................................................... page 32
  4.3. EC-DECAY OF 163HO ISOTOPE ................................................................................ page 33
  4.4. The Holmes Project ..................................................................................................... page 36
5. Holmium Implantation and Analysis of Gold thin films ..................................................... page 41
  5.1. Gold Thin Films Fabrication ........................................................................................ page 41
  5.2. Ho and Er Implantation and Characterization .............................................................. page 42
    5.2.1. Implantation Process .............................................................................................. page 42
    5.2.2. Thin Film Characterization Techniques ............................................................ page 46
6. Low Temperature Measurements ....................................................................................... page 62
  6.1. Experimental Setup ..................................................................................................... page 63
  6.2. Dilution Refrigerator .................................................................................................... page 64
  6.3. Electronics and signal readout .................................................................................... page 67
7. Conclusions and Perspectives ............................................................................................ page 70
1 Brief Introduction

The determination of the neutrino mass has important consequences. Considering only the conventional distinction between matter, composed of hadrons and leptons, and the forces described by bosons, an immediate observation is that neutrinos are the most common form of matter, about 330 neutrinos per cm$^3$ [8], while the baryon density of the universe is estimated to be $2\times10^{-7}$ per cm$^3$, considering the density of baryonic mass $4\times10^{-31}$ g/cm$^3$ [9] and baryons with the mass of the proton $1.67\times10^{-27}$ kg. The knowledge of the neutrino mass in cosmology is crucial for understanding the formation in large scale Universe, while in particle physics would help to make understandable the mechanism of mass production. Yet, the absolute mass of the three known types of neutrinos, electron, muon and tau neutrinos, is still unknown and a hot physics R&D topic.

The neutrino absolute mass measurement is a very difficult process, since this particle has a very small mass, and it can reach some eV, as obtained from tritium $\beta$-decay experiments [10]. Furthermore, neutrinos only interact through weak interaction or gravity, passing through matter without being affected by it. It is therefore necessary to achieve high precision experiments at the limits of current technology, able to achieve sensitivities of 0.2 eV. To implement this measure, the kinematic method is the traditional and most direct, since it does not depend on any model, and is achieved using $\beta$-decay. There are two different detector technologies to access this method, one uses as detector the spectrometer and the other the microcalorimeter. Presently, KATRIN is the next generation experiment that uses the spectrometer to measure the tritium beta decay [11] and MARE and HOLMES are the next generation experiments using the microcalorimeter to measure the $^{187}\text{Re}$ beta decay [12] and the $^{163}\text{Ho}$ electron capture decay [13], respectively.

This dissertation focused on the calorimetric technique used by MARE and HOLMES experiments. Monte Carlo simulations have showed that a microcalorimeter, that meets the criteria needed, can measure the neutrino mass with a sensitivity smaller than 1 eV [13], for an energy resolution of 1 eV and a pile-up resolving time of 1 $\mu$s. In a microcalorimetric approach, when the detector measures the radioactive source decaying ($\beta^\pm$ or by electron capture), the emitting ionizing radiation is equal to the total energy of the decay depressed by the one carried by the neutrino, which escapes. This energy is absorbed by the absorber, which transforms it into thermal energy, being then measured by the temperature sensor. The radioactive sources used must have the lowest Q-value as possible, in order to maximize the useful experimental statistics. Two isotopes have these characteristics: $^{187}\text{Re}$ with Q-value $\approx$ 2.5 keV [14] and $^{163}\text{Ho}$ with a Q-value still not well known, but that it is comprised between 2.2 and 2.8 keV [15].

There have been several experiments using $^{187}\text{Re}$. So far, the MANU experiment at Genoa [16] and the MIBETA experiment at Milano [17] are the only two experiments based on calorimetric measurement of rhenium beta decay. The MARE experiment is an update of MANU and MIBETA experiments, and explores the microcalorimetric technique using the beta decay of the 187 Rhenium isotope, aiming to measure an electron neutrino mass with sub-electronvolt sensitivity, $m_{\nu_e}\sim0.1\text{ eV}/c^2$. Due to its $43\times10^9$ years half-life [14], a parallel experiment is being studied with $^{164}\text{Ho}$. The HOLMES experiment was born from the MARE collaboration, and it will use the same detector technology as MARE, but a different isotope, which is $^{160}\text{Ho}$ and it decays through electron capture [18].

Microcalorimeters operating with Transition Edge Sensors (TES) are becoming the detectors of choice for different applications, such as in nuclear physics, astrophysics and astronomy [19, 20]. Superconducting TES are capable of imaging spectroscopy with high energy resolution, 2 eV FWHM at 6 keV, and this technology has already achieved a world record energy resolution of 1.8 eV FWHM at 6 keV in an 8×8 array [21].

The present work focused on the optimization of detector parameters, in particular heat capacity of the absorber material, which is one of the main parts of a microcalorimeter.

This manuscript is organized as follows: the second chapter presenting an brief introduction on neutrinos; the third chapter consisting on the operation theory of cryogenic microcalorimeters with transition edge sensors; the fourth chapter presenting a description of what has been done and
what is planned for both MARE and HOLMES experiments; the fifth and sixth chapters presenting the experimental results obtained during this work.
2 Brief Introduction on Neutrino Physics

Neutrino physics is playing a unique role in elucidating the properties of weak interactions. The neutrino studies are capable of providing new ingredients for future theoretical descriptions of the physics of elementary particles. Although impressive progress has been made in our understanding of neutrino interactions, many important issues remain to be settled, such as its absolute mass value.

It was expected, from the conservation of four-momentum, that the electron energy would be mono-energetic, equal to the difference in mass between nucleus father and daughter nucleus. However, it turned out that the electron always carried off less energy than expected, and instead of a discrete spectrum, there was a continuous distribution, as shown in Fig. 2.1. In this figure, the red line marks the expected electron energy, if only an electron was emitted and the blue line shows the observed electron energies.

![Electron Energy Spectrum for beta-decay of carbon-14](image)

This result, appeared to contradict conservation of energy, under the current assumption that beta decay is the simple emission of an electron from a nucleus. This problem was eventually resolved in 1930 by Wolfgang Pauli [23], who proposed the existence of a light neutral particle of spin 1/2 emitted alongside the electron in beta decay, which carries away the apparently missing energy. This explains the continuous spectrum, where the available energy is split between the electron and the undetected neutral particle. Enrico Fermi, in 1934, called it a neutrino, "little neutral one", within the formulation of his theory for beta decay [24]. The success of this theory established the existence of the neutrino and due to their extremely elusive properties, such as, close to zero mass and no charge, it was a challenging task for scientists to design experiments to detect them.

The advent of nuclear fission in the 1930s and 1940s offered an unprecedentedly intense source of (anti-)neutrinos, which for the first time made possible the experimental detection of these elusive particles [25]. Nuclear fission produces neutrinos, since fission of heavy elements makes isotopes that have too many neutrons to be stable. In this way, these isotopes find it energetically favourable to convert the excess neutrons into protons, resulting in a cascade of beta decays.

In the mid-1950s, the existence of an electron neutrino was experimentally confirmed for the first time by Fred Reines and Clyde Cowan [26]. The Cowan-Reines neutrino experiment was
based on the observation of the inverse beta decay reaction, produced by anti-neutrino interactions. In particular, when a proton interacts with an anti-neutrino produces a neutron and a positron:

\[ \bar{\nu}_e + p \to n + e^+ \quad (2.1) \]

The outgoing positron quickly reacts with an electron releasing two detectable gamma rays. Since the coincident detection of the pair annihilation gammas was not quite proof of neutrino detection by this reaction, Reines and Cowan tried to detect the neutron from the reaction by putting cadmium chloride into a detector tank. When absorbing a neutron, \(^{108}\text{Cd}\) produces an excited state of \(^{109}\text{Cd}\) which subsequently emits a gamma ray through the reaction:

\[ n + ^{108}\text{Cd} \to ^{109}\text{Cd}^* \to ^{109}\text{Cd} + \gamma \quad (2.2) \]

The experimental arrangement was such that the additional gamma from this process was detected within about \(5 \times 10^{-6}\) seconds after the \(e^+ - e^-\) pair annihilation gammas [26]. This provided a distinctive signature for the neutrino reaction, the gamma pair in coincidence plus another gamma within 5 \(\mu\)s.

The next advancement occurred in 1962, when muon neutrinos were discovered at Brookhaven National Lab via observations of muonic decay [27]. The existence of the tau neutrino, the last lepton predicted by the Standard Model, was confirmed in 2001 by the “Directed Observation of the NU Tau” (DONUT) experiment at Fermi lab [28].

Since the discovery of the muon neutrino, it appeared to be clear that neutrinos associated with electrons and muons, called electron-neutrinos, \(\nu_e\), and muon-neutrinos, \(\nu_\mu\), respectively, were different, and that the law of conservation of lepton number was obeyed separately for electrons and muons. These properties were well described by a model invented almost simultaneously by Lee and Yang, Landau, and Salam [29], in which the neutrinos had exactly zero mass.

In the late 1960s, astrophysicists Raymond Davis and John N. Bahcall headed an experiment, the first to successfully detect and count solar neutrinos, called The Homestake experiment [30, 31]. Its purpose was to collect and count neutrinos emitted by nuclear fusion taking place in the Sun. The rate at which the detector should capture neutrinos, turned out to be only one third of the theoretical value. The discrepancy between the predicted and measured rates of neutrino detection was later found to be due to neutrino "flavour" oscillations.

The Homestake experiment was followed by other experiments with the same purpose, such as Kamiokande in Japan, SAGE in the former Soviet Union, GALLEX in Italy, Super Kamiokande, also in Japan, and SNO (Sudbury Neutrino Observatory) in Ontario, Canada. SNO was the first detector able to detect neutrino oscillation [32]. The results of the experiment, published in 2002, revealed that of the three “flavours” between which neutrinos are able to oscillate, Davis’s detector was sensitive to only one. These oscillations experiments are a prove that neutrinos do have mass, and that neutrinos produced in association with electrons can subsequently interact as muon-neutrinos.

The masses of neutrinos are extremely small, and therefore the oscillation between different types, known as flavours, is very small. Only very sensitive experiments can have allow the demonstration of these subtle effects.

Neutrino mass is arguably the most important discovery in particle physics in the last quarter century. Exploring the spectrum of the neutrinos and their properties under the CP symmetry will allow the understanding of neutrinos and their role in the Universe.

Neutrinos are the most abundant constituents of matter, around 300 neutrinos per \(cm^3\), and also one of the least understood. These particles were produced in the Big Bang and, are spin-1/2 fermions. They are electrically neutral, and interact with other particles only through the weak interaction and gravity. Neutrinos are fundamental particles capable of traveling with velocities close to the speed of light and almost undisturbed through baryonic matter.
Although, it is now known that neutrinos have a non-zero mass value, both through oscillations experiments and through beta-decay spectrum measurements [33], its absolute value has not been determined yet.

Neutrino mass is a phenomenon that cannot be understood within the Standard Model, since from neutrino oscillations some effects of Lorentz violation can be seen [34].

The nature of the neutrino is not settled and it may be either a Dirac particle, where in this case the neutrino will have a distinct anti-particle or a Majorana particle, where the neutrino is its own anti-particle [35]. Non-oscillation experiments, like cosmological observations, searches for neutrinoless double beta decay, and high precision measurements of single beta decay, are being carried out for the experimental determination of neutrino mass [36, 37]. Cosmological research is sensitive to the sum of the neutrino masses [38], and so neutrinoless double beta decay has been the only known technique to determine whether neutrinos are Dirac or Majorana particles [39].

To date the most sensitive upper limit of electron neutrino mass has been obtained by direct observation of decay kinematics in single $\beta$-decay process [40]. Currently two independent methods, spectrometric and calorimetric measurement of $\beta$-spectrum, have been adopted for the electron neutrino mass measurement from the end-point study of $\beta$-decay [41].

2.1 Beta Decay

Natural radioactivity consists in the spontaneous emission, by some nuclei, of ionizing radiation. Three major radioactive emissions are the $\alpha$-rays, consisting of $^4$He nuclei, $\beta$-rays, consisting in the emission of electrons or positrons, and $\gamma$-rays, which are a type of electromagnetic radiation. In the first two cases, the radio-nuclei that decay spontaneously transform into atoms of another element, in the third case there is only a modification of the nuclear orbitals that tend to a more stable configuration.

The $\beta$-decay is a manifestation of a fundamental interaction in nature: the weak interaction. This decay is a radioactive decay in which a proton in a nucleus is converted into a neutron (or vice-versa). In the process the nucleus emits a beta particle (either an electron or a positron) and quasi-massless particle, the neutrino. Beta decay is an excellent candidate to study the properties of the neutrino, since it is a natural source of this particle.

The radioactive nucleus decays into a isobaric nucleus, with a charge difference of $Z = \pm 1$. This can occur through the emission of an electron with an atomic charge variation of $Z = +1$ (decay type $\beta^-$), through the emission of a positron with an atomic charge variation of $Z = -1$ (decay type $\beta^+$), or through the capture of an electron with an atomic charge variation of $Z = -1$ (decay by electron capture).

Beta decay takes place, since the daughter nucleus has a lower mass than the parent, and therefore the decay is energetically favoured. The electron capture decay, EC decay, referred to as "inverse beta decay," as the basic process, mediated by the weak force, is the same.

The measured energy spectrum for the emitted electrons is continuous, with maximum upper limit equal to the energy available to the process, defined Q-value or end-point. An unstable atomic nucleus with an excess of neutrons may undergo $\beta^-$ decay, while an unstable atomic nuclei with an excess of protons may undergo $\beta^+$ decay, or inverse beta decay. Unlike beta minus decay, beta plus decay cannot occur in isolation, since it requires energy, due to the fact of the mass of the neutron being greater than the mass of the proton. Beta plus decay can only happen inside nuclei when the absolute value of the binding energy of the daughter nucleus is higher than that of the parent nucleus. The difference between these energies goes into the reaction of converting a proton into a neutron, a positron and, a neutrino and into the kinetic energy of these particles. That is why the beta plus and the beta minus decay spectra are slightly different, due to Coulomb repulsion or attraction from the nucleus, and the maximum energy available for the beta plus spectrum is higher, as shown in Fig. 2.2.
Fermi, in his theory [24], does not consider the $\beta$-decay as an electron loss by the nucleus, but rather a simultaneous creation of electron-neutrino together with a corresponding change in the charge of a nucleon. The different types of beta decay, including the EC process, are described by the following equations:

\[ n \rightarrow p + e^- + \bar{\nu}_e \quad \text{$\beta^-$ decay} \]  \hspace{1cm} (2.3)

\[ n \rightarrow p + e^+ + \nu_e \quad \text{$\beta^+$ decay} \]  \hspace{1cm} (2.4)

\[ p + e^- \rightarrow n + \nu_e \quad \text{EC decay} \]  \hspace{1cm} (2.5)

where $e^-$ is the electron, $e^+$ the positron, $\nu_e$ and $\bar{\nu}_e$ are the neutrino and anti-neutrino, respectively.

The three main processes of $\beta$-decay describing the transformation of an unstable nucleus into its isobar nuclei are:

\[ N(Z, A) \rightarrow N(Z + 1, A) + e^- + \bar{\nu}_e \]  \hspace{1cm} (2.6)

\[ N(Z, A) \rightarrow N(Z - 1, A) + e^+ + \nu_e \]  \hspace{1cm} (2.7)

\[ N(Z, A) + e^- \rightarrow N(Z - 1, A) + \nu_e \]  \hspace{1cm} (2.8)

where $N(Z, A)$ indicates a radio-nucleus of atomic weight $A$ and atomic charge $Z$.

The Q-value of the reaction provides a measure of the energy released in the process. If this value is positive then the reaction is permitted, otherwise is prohibited. In general, the Q-value of a reaction is defined as the change between the final, $T_f$, and the initial, $T_i$, kinetic energy of the system, such as:

\[ Q = T_f - T_i \]  \hspace{1cm} (2.9)
Considering the initial core stationary in the laboratory frame, then $T_i = 0$, if the decay 
occurs, $T_f$ must be positive, leading to a positive $Q$-value.

For $\beta^-$ and $\beta^+$ decays the $Q$-value is not exactly the mass difference between the final state 
and the initial one, as an electron or a positron can be produced. In fact, one needs to take into 
account the mass and the binding energy of an atom of charge $Z$. Defining the mass difference 
between the parent atom and daughter atom as:

$$\Delta M_{\beta^\pm} = M(Z, A) - M(Z \pm 1, A)$$  \hspace{1cm} (2.10)$$

where the mass and the binding energy of the atomic electrons are included.

Therefore, from Eq. 2.10, the $Q$-value of a $\beta^-$-decay reaction will be given by:

$$Q_{\beta^-} = [M(Z, A) - M(Z + 1, A)]c^2$$  \hspace{1cm} (2.11)$$

The electron emitted in the $\beta^-$-decay can be used to compensate the additional electron 
required to produce the neutral atom in the decay.

In a $\beta^+$-decay, one has:

$$Q_{\beta^+} = [M(Z, A) - M(Z - 1, A)]c^2 - 2m_e c^2$$  \hspace{1cm} (2.12)$$

where the $2m_e c^2$ term is due to the creation of a positron and an electron emitted from the nucleus 
to balance the charge.

In the case of EC decay, one has:

$$Q_{EC} = [M(Z, A) - M(Z - 1, A)]c^2$$  \hspace{1cm} (2.13)$$

Furthermore, rewriting the $Q$-value for EC as a function of $\beta^+$, such as:

$$Q_{EC} = Q_{\beta^+} + 2m_e c^2$$  \hspace{1cm} (2.14)$$

The nucleus decays if the $Q$-value is positive. From Eq. 2.14, one can observe that if a nucleus 
decays as $\beta^+$, where $Q_{\beta^+} > 0$, it also decays by electron capture, since its $Q$-value is for sure greater 
than zero.

The kinetic energy, equal to $Q$, is equal to the energy carried by the neutrino plus the one 
carried by the electron, where any recoil of the massive nucleus is neglected, as the electron energy 
is much smaller than the mass and recoil energy of the nucleus.

The $Q$-value is the energy available in the reaction to be divided between the electron and 
antineutrino or positron and neutrino, and the mass of the neutrino, where the small kinetic energy 
transferred to the daughter nucleus is neglected. For the three types of decay mentioned above it 
can be written:

$$Q_{\beta^-} = \Delta M c^2 = T_{\nu_e} + T_{\bar{\nu}_e} \hspace{1cm} \beta^- \text{ decay}$$  \hspace{1cm} (2.15)$$

$$Q_{\beta^+} = \Delta M c^2 - 2m_e c^2 = T_{\nu_e} + T_{\bar{\nu}_e} \hspace{1cm} \beta^+ \text{ decay}$$  \hspace{1cm} (2.16)$$

$$Q_{EC} + m_e c^2 - B_e = \Delta M c^2 + m_e c^2 - B_e = T_{\nu_e} \hspace{1cm} \text{EC decay}$$  \hspace{1cm} (2.17)$$

where $T_{\nu_e}$ and $T_{\bar{\nu}_e}$ are respectively the energies of the electron and the positron, $m_e$ is the electron 
mass and $T_{\nu_e}$ and $T_{\bar{\nu}_e}$ are respectively the energies of the neutrino and antineutrino. In the case of EC 
decay, $B_e$ indicates the binding energy of the captured atomic electron, which is typically of the 
order of tens of eV, and therefore, in general, can be negligible compared to the value of the energies 
into play.
Besides energy, there are other conserved quantities, such as:

- Momentum: this quantity is also shared between the electron and the neutrino. Thus the observed electron momentum ranges from zero to a maximum possible momentum transfer.
- Angular momentum: both the electron and the neutrino have spin \( \frac{1}{2} \).
- Charge: the creation of a proton is for example always accompanied by the creation of an electron.
- Particle number: the total number of particles is not conserved, the beta particles and neutrinos are created. However the number of massive, heavy particles, called baryons, composed of 3 quarks, is conserved.
- Lepton number: is conserved. Leptons are fundamental particles, including the electron, muon and tau, as well as the three types of neutrinos associated with these particles. The lepton number is +1 for these particles and -1 for their antiparticles. Then an electron is always accompanied by the creation of an antineutrino, e.g., to conserve the lepton number (initially zero).

It turns out that parity is not conserved in this decay. This hints to the fact that the interaction responsible violates parity conservation, so it cannot be the same interactions already studied, such as electromagnetic and strong interactions. The helicity is the projection of the spin on the momentum of the particle. In fact, neutrino has negative helicity, meaning that the directions of spin and motion are opposite, and the neutrino is called left-handed, while the anti-neutrino has positive helicity, where the direction of its spin is the same as the direction of its motion, and the neutrino is called right-handed.

The properties of beta decay can be understood by studying its quantum mechanical description via Fermi’s Golden rule [43]. Quantum field theory gives a unification of electromagnetic and weak force (electro-weak interaction) with one coupling constant \( e \). The interaction responsible for the creation of the electron and neutrino in the beta decay is called the weak interaction and its one of the four fundamental interactions, together with gravitation, electromagnetism and the strong interaction that keeps nucleons and quarks together. One characteristic of this interaction is parity violation.

### 2.1.1 Density of States

Knowing the density of states, is possible to calculate how many electrons are emitted in the beta decay with a given energy. This will be proportional to the rate of emission calculated from the Fermi Golden Rule, times the density of states. For \( \beta \)-decay, in the calculation of the density of states, one needs to take into account the fact that there are two types of particles, the electron and neutrino, as products of the reaction, and both can be in a continuum of possible states. Then the number of states in a small energy volume is the product of the electron and neutrino’s states, such as:

\[
d^2N_e = dN_e dN_\nu \tag{2.18}
\]

The two particles share the \( Q \) energy:

\[
Q_{\beta \pm} = Q_\beta = T_e + T_\nu = T_e + T_\nu \tag{2.19}
\]

where from now on it will be use indistinctly the symbol \( e \) for electron and positron, and the symbol \( \nu \) for neutrino and antineutrino.

Assuming that the mass of the neutrino is zero, since it is much smaller than the electron mass and then the kinetic mass of the neutrino itself, then the following relativistic expression can be used:
where \(c\) is the speed of light in vacuum, \(3 \times 10^8\) m/s, and \(p_v\) is the neutrino’s momentum.

For the electron, its energy is expressed as:

\[
E^2 = p^2 c^2 + m^2 c^4 \rightarrow E_e = T_e + m_e c^2
\]

where \(T_e = \sqrt{p_e^2 c^2 + m_e^2 c^4} - m_e c^2\) and \(p_e\) is the electron’s momentum.

From Eq. 2.19, the kinetic energy of the neutrino can be written as a function of the electron’s, such as:

\[
T_v = Q_\beta - T_e
\]

The number of states for the electron can be calculated from the quantized momentum, under the assumption that the electron state is a free particle \((\psi \sim e^{ik \cdot r})\) in a region of volume \(V = L^3\):

\[
dN_e = \left(\frac{L}{2\pi}\right)^3 4\pi k^2 dk = \frac{4\pi v}{(2\pi \hbar)^3} p_e^2 dp_e
\]

where the relationship between the momentum and wave number, \(p = \hbar k\), was used and \(\hbar\) is the reduced Planck constant.

The number of states for the neutrino is calculated in the same way as for the electron and is given by:

\[
dN_v = \frac{4\pi v}{(2\pi \hbar)^3} p_v^2 dp_v
\]

At a given momentum/energy value for the electron, the density of states, \(\rho(p_e)d\rho_e\), can be written as:

\[
\rho(p_e)d\rho_e = dN_e \frac{dN_v}{dT_v} = 16\pi^2 \frac{v^2}{(2\pi \hbar)^3} p_e^2 dp_e \frac{dp_v}{dT_v}
\]

From Eqs 2.20 and 2.22, one gets \(dT_v/dp_v = c\) and \(p_v = (Q_\beta - T_e)/c\). Substituting these terms in Eq. 25, one obtains:

\[
\rho(p_e)d\rho_e = \frac{v^2}{4\pi^* \hbar^* c^2} [Q_\beta - T_e]^2 p_e^2 dp_e
\]

Using \(T_e = \sqrt{p_e^2 c^2 + m_e^2 c^4} - m_e c^2\) from Eq. 2.21, Eq. 2.26 becomes:

\[
\rho(p_e)d\rho_e = \frac{v^2}{4\pi^* \hbar^* c^2} [Q_\beta - \sqrt{p_e^2 c^2 + m_e^2 c^4} - m_e c^2]^2 p_e^2 dp_e
\]

For a massive neutrino, the energy for the neutrino in Eq. 2.20 is replaced by:

\[
E_v = (m_v^2 c^4 + p_{2v}^2 c^2)^{1/2} \rightarrow E_v = T_v + m_v c^2
\]

where \(T_v = \sqrt{p_v^2 c^2 + m_v^2 c^4} - m_v c^2\) and \(p_v\) is the neutrino’s momentum.

The neutrino’s momentum is given by:

\[
p_v = \frac{\sqrt{E_v^2 - m_v^2 c^4}}{c}
\]
Therefore, to calculate the density of states in this case, the term \( \frac{dp_v}{dE_v} \) will be given by:

\[
\frac{dp}{dE_v} = \frac{E_v}{c \sqrt{E_v^2 - m_v^2 c^4}} = \frac{Q_\beta - E_e}{c \sqrt{(Q_\beta - E_e)^2 - m_v^2 c^4}}
\]  

(2.30)

where \( E_v = Q_\beta - E_e \).

Substituting Eq. 2.29 in Eq. 2.25, the density of state for a massive neutrino becomes:

\[
\rho(p_e) d\rho_e = 16\pi^2 \frac{v^2}{(2\pi \hbar \epsilon)^3} \frac{1}{c^3} (Q_\beta - E_e) \sqrt{(Q_\beta - E_e)^2 - m_v^2 c^4} p_e^2 dp_e
\]

(2.31)

Both Eqs 2.26 and 2.31 can be used to calculate how many electrons are emitted in the beta decay with a given energy, meaning the decay rate, for a massless or massive neutrino, respectively, through Fermi’s Golden Rule, which will be explained in the next section.
2.1.2 Decay Rate

For situations where the transition probability is constant in time, it is usually expressed in a relationship called Fermi’s golden rule. In general conceptual terms, a transition rate depends upon the strength of the coupling between the initial and final states of a system and upon the number of ways the transition can happen, i.e., the density of the final states. A transition will proceed more rapidly if the coupling between the initial and final states is stronger. This coupling term is traditionally called the “matrix element” of the transition.

Fermi’s Golden Rule is given by:

\[ W = \frac{2\pi}{\hbar} |V_{ij}|^2 \rho(E) \]  

(2.32)

where \( \rho(E) \) is the density of states.

Eq. 2.32, for beta decay, is the product of three terms:

- the Statistical factor, arising from the density of states calculation;
- the Fermi function, accounting for the Coulomb interaction, attraction/repulsion, between the emitted beta and the final state nucleus;
- between the nucleus and the electron, since is not only the weak interaction that is at play, \( F(Z, Q) \);
- the Transition amplitude, from the Fermi Golden Rule, \( |V_{ij}|^2 \).

These three terms are the three ingredients that determine the spectrum and decay rate of beta decay processes. \( F(Z, Q) \), called the Fermi function, that takes into account the shape of the nuclear wavefunction and in particular it describes the Coulomb attraction or repulsion of the electron or positron from the nucleus. Thus, \( F(Z, Q) \) is different, depending on the type of decay. \( \rho(E) \), and thus the decay rate, is obtained by summing over all possible states of the beta particle, as counted by the density of states. From Eq. 2.32, is possible to calculate the spectrum of the emitted beta particles, the electron or positron. Thus, in practice, one needs to integrate the density of states over all possible momentum of the outgoing electron/positron. For a zero mass neutrino, using Eq. 2.26, upon integration over \( \rho(E) \) one obtains:

\[ \rho(E) = \frac{\nu^2}{4\pi^4\hbar^6c^3} \int_0^{p_{\text{max}}} dp_e [Q_\beta - T_e]^2 p_e^2 \approx \frac{\nu^2}{4\pi^4\hbar^6c^3} \frac{(Q_\beta - mc^2)^5}{30c^3} \]  

(2.33)

where the relativistic limit for a high electron speed was taken, \( T_e \approx pc \).

The decay rate can then be written as:

\[ W = \frac{2\pi}{\hbar} \frac{g^2}{V^2} |M_{np}|^2 F(Z, Q_\beta) \frac{\nu^2}{4\pi^4\hbar^6c^3} \frac{(Q_\beta - mc^2)^5}{30c^3} = G_F^2 |M_{np}|^2 F(Z, Q_\beta) \frac{(Q_\beta - mc^2)^5}{60\pi^3 \hbar (hc)^6} \]

where the matrix element \( V_{ij} = (g/V)M_{np} \) and, due to Fermi’s Golden Rule, \( |M_{np}|^2 \rightarrow |M_{np}|^2 F(Z, Q_\beta) \). The constant \( G_F = (1/\sqrt{2\pi^3})(gm^2c/hc^3) \), and it gives the strength of the weak interaction. Comparing to the strength of the electromagnetic interaction, as given by the fine constant, \( \alpha = e^2/\hbar c \sim 1/137 \), the weak interaction is much smaller, with a constant of \( \sim 10^{-6} \).

The differential decay rate is given by:

\[ \frac{dW}{dp_e} = \frac{2\pi}{\hbar} |V_{ij}|^2 \rho(p_e) \propto F(Z, Q_\beta) [Q_\beta - T_e]^2 p_e^2 \]  

(2.34)

The square root of this quantity is then a linear function in the neutrino kinetic energy, \( Q_\beta - T_e \):
\[
\frac{dW}{dp_e} \frac{1}{p_e^2 F(Z, Q_{\beta})} \propto Q_{\beta} - T_e
\]  
(2.35)

This is the Fermi-Kurie relation for a zero mass neutrino in a \( \beta \)-decay. The Fermi-Kurie plot, is a graph used in the study of beta decay developed by Franz N. D. Kurie. This plot is a straight line, in which the square root of the number of beta particles whose momenta or energy lie within a certain narrow range, divided by the Fermi function, is plotted against beta-particle energy. The interception of the Kurie plot with the energy-axis, the x-axis, corresponds to the maximum energy imparted to the electron/positron, the decay’s Q-value. With this plot one can find the limit on the absolute neutrino mass value.

For a massive neutrino the differential decay rate will be given by:

\[
\frac{dW}{dp_e} \propto F(Z, Q_{\beta})(Q_{\beta} - E_e) \sqrt{(Q_{\beta} - E_e)^2 - m_{\nu}^2 c^4 p_e^2}
\]  
(2.36)

The influence of neutrino mass on the beta spectrum near the end-point can best be seen in a linearized spectrum in the following way:

\[
\frac{dW}{dp_e} \frac{1}{p_e^2 F(Z, Q_{\beta})} \propto \sqrt{(Q_{\beta} - E_e) \sqrt{(Q_{\beta} - E_e)^2 - m_{\nu}^2 c^4}}
\]  
(2.37)

The maximum energy of the electron and the neutrino, respectively, will be:

\[
E_{e}^{\text{max}} = Q_{\beta} - m_{\nu} c^2
\]  
(2.38)

\[
E_{\nu}^{\text{max}} = Q_{\beta} - m_e c^2
\]  
(2.39)

The total kinetic energy released in \( \beta \)-decay is the difference in rest energy between the initial nucleus and the end-products, and so, when performing calculations of a \( \beta \)-spectrum. Using Eqs 2.38 and 2.39, is possible to derive the electron and neutrinos kinetic energies such as:

\[
T_{e}^{\text{max}} = E_{e}^{\text{max}} - m_e c^2 = Q_{\beta} - m_{\nu} c^2 - m_e c^2
\]  
(2.40)

\[
T_{\nu}^{\text{max}} = E_{\nu}^{\text{max}} - m_{\nu} c^2 = Q_{\beta} - m_e c^2 - m_e c^2
\]  
(2.41)

From Eqs 2.40 and 2.41, one sees that the kinetic energy of both electron and neutrino will vary continuously in the same interval between \( 0 < T_{e,\nu} < Q_{\beta} - m_e c^2 - m_{\nu} c^2 \).

The \( \beta \) spectrum in the case where \( m_{\nu} = 0 \), given by Eq. 2.35, deviates from the \( \beta \) spectrum for \( m_{\nu} \neq 0 \), given by Eq. 2.37, in the lower \( (p_e \rightarrow 0) \) and upper \( (E_e = E_e^{\text{max}} = (Q_{\beta} - E_e)) \) limits of electron’s momentum.

The plot in Fig. 2.3 is given by the following equations, derived from Eqs 2.37 and 2.39, which have a different performance when the neutrino is considered massive or massless:

\[
\frac{dW}{dp_e} \frac{1}{p_e^2 F(Z, Q_{\beta})} = \begin{cases} 
C(Q_{\beta} - T_e) & m_\nu = 0 \\
C \sqrt{(Q_{\beta} - E_e) \sqrt{(Q_{\beta} - E_e)^2 - m_{\nu}^2 c^4}} & m_\nu \neq 0
\end{cases}
\]  
(2.42)

Measuring the electrons with energies near the end-point, Q-value, with the correct resolution, it is possible to determine the corresponding neutrino mass.
From Eq. 2.42, it can be seen that appreciable sensitivity to neutrino mass exists only near the end-point of the spectrum. The spectrum for a massive neutrino terminates abruptly, with infinite slope, at an energy \( Q_\beta - m_\nu \), manifestly distinguishing it from the spectrum for the case of a massless neutrino. Experimentally, it is required an electron energy \( E_e \) near \( Q_\beta \), so that \( Q_\beta - E_e \equiv \Delta E \) is of the order of \( m_\nu \).

For a low energy electron decay with maximum kinetic energy \( E_e = Q_\beta - m_\nu \ll Q_\beta \), which is the region of interest, the fraction of decays in the interval \( \Delta E \) is given by approximately \( (2\Delta E/E_e)^3 \) [12]. \( \Delta E \) is thus, the size of the relevant energy interval of the region near the end-point which is approximately \( 3m_\nu e \), but is limited by detector energy resolution [44]. The fraction \( (\Delta E/Q)^3 \) becomes rapidly smaller for beta decays with higher \( Q \) values, such as \(^{63}Ni (Q = 67 \text{ eV})\) or \(^{35}S (Q = 167 \text{ eV})\) [45]. It is, therefore, imperative to select a candidate with low \( Q \) value. In addition, another important criterion in selecting a decay, is that its lifetime should be relatively short, which means atoms decay more rapidly, making more data available. Tritium, for example is a good source since it has a reasonably short lifetime, 12.4 years [46]. The longer the lifetime, the higher the isotope concentration required for equal decay rates.

The evaluation of the detector sensitivity, energy resolution, efficiency, rise time and dead time is therefore crucial for the neutrino mass measurement. For low decay rates at the end point region the analysis of the Kurie plot is affected by the background due to cosmic rays and environmental radioactivity. In this way, the very low count rate in the region of interest demands a very low background level of 10 mHz to reach a design sensitivity of 200 meV and a detector with very high energy resolution of 1 eV [47]. The statistical fitting of the measured and calculated shape of the spectra [44] gives \( m_{\nu e}^2 \).
2.1.3 Energy Spectrum of Electron Capture Decay

The suggestion that positron emitters might decay by the alternative process of electron capture was first advanced by Yukawa [48] from considerations based on the Fermi theory of beta-ray emission.

On the basis of Dirac's theory, however, the positron is merely the "hole" left in the continuum of negative energy electrons when one of these electrons is given a positive energy by the addition of at least $2m_e c^2$ [49]. The proton in Eq. 2.4 does not transform into a neutron and positron, but rather captures a negative energy electron, and turns into a neutron, leaving the hole in the negative energy sea, or positron. Eq. 2.4 can be written as Eq. 2.5: when the energy difference between parent and daughter nucleus is less than $2mc^2$, the $\beta^+$-decay cannot happen unless a proton could capture an ordinary electron. Since there are many cases of negative beta decay with an energy released of less than this value [49] it is natural to suppose that there would be excited nuclei not able to emit positrons, due to the fact of not be allowed on energetic grounds. Yukawa suggested [48], that in these cases, the decay would proceed by the capture of an orbital electron. In addition, Yukawa and others [50, 51, 52, 53], extended the calculations, that even when there was enough energy to create a pair by the $\beta^+$-decay, a certain fraction of the excited nuclei would decay by electron capture. The branching ratio of the two processes was found to depend on the energy available, the spin change involved, and the nuclear charge (density of electrons at the nucleus). Electron capture should become more probable as the energy decreases, and as the spin change, atomic number and half-life increase.

X-rays and Auger electrons emitted in the de-excitation of the ionized daughter provide more readily detectable signals of the capture process. Alvarez [54] first gained experimental evidence for the existence of nuclear electron capture by detecting Ti K X-rays emitted in the decay of $^{48}$V.

In the electron capture decay no beta particle is emitted, but only an electron neutrino. Unlike $\beta^+$ emission, an inner atomic electron is captured by a proton in the nucleus. This type of decay is therefore analogous to positron emission, and it happens in all positron-emitters. Electron capture is the only type of decay that is allowed in proton-rich nuclides which do not have sufficient energy to emit a positron and a neutrino, but may still reach a lower energy state by the equivalent process of electron-capture and neutrino-emission.

The mass difference between the parent and daughter neutral atoms is given by:

$$\Delta M = W_0 + 1$$  \hfill (2.43)

where the natural units $h = m_e = c = 1$, where used.

The quantity $W_0$ is equal to:

$$W_0 = Q_{EC} - B_e = Q_{EC} - \Delta|\Sigma_j E_j|$$  \hfill (2.44)

where $Q_{EC}$ is the energy difference between the parent nucleus $(A,Z)$ and the daughter nucleus $(A,Z - 1)$ and the amount $\Delta|\Sigma_j E_j|$ represents the total change in electron binding energy between the parent and daughter atoms. This change arises since, due to the decay, the charge of the daughter nucleus differs from that of the parent nucleus from $Z = -1$, presenting a different orbital configuration of electrons.

Let $E_j'$ be the binding energy of the captured electron in the $j$ shell of the daughter atom. Due to imperfect atomic wave-function overlap the daughter atom's electronic excitation energy, $E_{exc}$, will exceed $|E_j'|$ by an amount denoted by $E_R$. The average of this rearrangement energy, $E_R$, taken over many atoms, is small (of the order of a few eV), but in those individual transitions in which substantial internal ionization occurs, $E_R$ can be quite significant. Neglecting the energy of atomic recoil from neutrino emission, the neutrino energy is:
\[ E_\nu = W_0 + 1 - E_{exc} = Q_{EC} - \Delta |\Sigma_j E_j| + 1 - |E'_j| - E_R \tag{2.45} \]

where \( E_{exc} = |E'_j| + E_R \).

As the neutrino interacts weakly, the detectable energy of this decay is \( E_{exc} \). The atomic excitation energy is released, after the capture event, in a cascade of Auger and radioactive transitions, except for energy carried by internal ionization.

The energy threshold for electron capture from orbital \( j \) is:

\[ Q_{EC} \geq -1 + \Delta |\Sigma_j E_j| + |E'_j| + E_R \tag{2.46} \]

Positron emission is energetically possible, and competes with orbital electron capture if \( W_0 \geq 1 \), or

\[ Q_{EC} \geq 1 + \Delta |\Sigma_j E_j| + E_R \tag{2.47} \]

The entire atom is transformed in electron-capture decay and is reflected in the effect of imperfect atomic wave-function overlap on the transition rate [55]. Furthermore, atomic transitions such as internal ionization can take place as an integral part of the radioactive decay, quite distinct from the Auger and X-ray cascade through which the daughter atom is subsequently de-excited.

The neutrino momentum is given by:

\[ p_\nu = W_0 + W'_j \tag{2.48} \]

where \( W_0 \) is the total transition energy between initial and final states, and \( W'_j = 1 - |E'_j| \), denotes the energy of the bound electron (in the daughter atom), where \( E'_j \) is the binding energy of the electron.

A complete description of the initial and final states must include the electrons of the atomic cloud. Since the nuclear charge and the number of electrons are different in the initial and final states, the atomic-electron wave functions of these two states are not orthogonal, and the overlap between them is not perfect.

Substituting Eq. 2.48 in Eq. 2.45, one can write the neutrino energy as a function of its momentum such as:

\[ E_\nu = p_\nu - E_R \tag{2.49} \]

The decay by electron capture involve electrons from different atomic shells, the decay rate will be given by the sum of the individuals decay rates from each captured electron. Through a series of physical considerations and mathematical simplifications [56], it is obtained that the total capture probability from all atomic shells for a massless neutrino, \( \lambda_{EC} \), is given by:

\[ \lambda_{EC} = \frac{g_\beta^2}{2\pi^2} \sum_j n_j C_j f_j \tag{2.50} \]

where \( g_\beta \) is the \( \beta \)-decay coupling constant which is related to the universal weak coupling constant, \( j \) indicates all the shells on which is possible can capture an electron including subshells, \( n_j \) is the occupation number related to the electrons in the shell \( j \), which equals 1 for closed shells and for partially filled shells is equal to the relative occupation number of the electrons in the shell, and the quantity \( C_j \) corresponds to the shape factor of \( \beta \)-decay.

The function \( f_j \) in Eq. 2.50, which corresponds to the integrated Fermi function of \( \beta \)-decay, has the form:

\[ f_j = \left( \frac{\pi}{2} p_j^2 \beta_j^2 B_j \right) \tag{2.51} \]
where $B_j$ factor is an atomic correction for electron exchange and overlap [56], and $\beta_j$ is the Coulomb amplitude of the electron radial wave function, essentially the modulus of the wave function at the origin, determined by the shape of the charge distribution of the surrounding atomic electrons.

The spin and parity of the nuclei involved in EC capture obey the selection rules $\Delta J = 0, 1$ and $\pi_i \pi_f = \pm 1$ [56], respectively, where $\Delta J = |J_i - J_f|$, is the variation of the total angular momentum between the initial and final states, and $\pi_i$ and $\pi_f$ are respectively the parity of the initial and final states. In this way, a nucleus, $N(Z, A)$, can decay into a nucleus, $N(Z - 1, A)$, only if it satisfies the selection rules.

Due to the fact of electron-capture rate is essentially proportional to the electron density at the nucleus, different chemical environments or other macroscopic perturbations (pressure, temperature, etc.) can affect the decay constant. Such effects are most noticeable in capture from outer electrons shells.

Furthermore, for a massive neutrino, the decay rate is:

$$\lambda_{EC} = \frac{g_\beta^2}{4\pi} \sum_j n_j C_j \beta^2_j B_j (Q - E'_j) \left[ (Q - E'_j)^2 - m_\nu^2 \right]^{1/2} \tag{2.52}$$

where $Q$ is defined as $Q = Q_{EC} - \Delta |\Sigma_j E_j| + 1$.

Eq. 2.52, represents therefore, the total capture rate from all shells can be obtained by summing over the possible levels of the captured electrons [56].

The spectrum resulting from the EC decay will, therefore, consist of a series of peaks typical of the ionization energies of the electrons belonging to the daughter atom. The atomic levels have a finite (albeit often small) natural width, $\Gamma$, typical of the electronic orbital captured, and therefore the lines have a Breit-Wigner resonance form, due to the fluorescence of the daughter excited atom.

The spectrum is continuous with evident peaks, and if the Q-value of the decay has a value close to one of the possible ionization energies $E'_j$, the rate near the end-point, where the effects of the neutrino mass are apparent, will increase due to a high increase in the resonance [57].

The spectrum of "calorimetric" energy or de-excitation energy, $E'_j$, is provided by the following function [58]:

$$\frac{d\lambda_{EC}}{dE'_j} = \frac{g_\beta^2}{4\pi^2} (Q - E'_j) \sqrt{(Q - E'_j)^2 - m_\nu^2} \sum_j n_j C_j \beta^2_j B_j \left[ \frac{\Gamma_j}{2\pi (E'_j - E_j)^2 + \Gamma_j^2} \right] \tag{2.53}$$

Taking the integral of Eq. 2.53, allows one to determine the transition probabilities for the different peaks, where $E'_j$ is, to a less than a small correction, the binding energy of the captured electron.

The probability of electron capture, $\lambda_{\nu} = \lambda_{\nu} / E'_j$, becomes much more sensitive to a neutrino mass, $m_\nu \neq 0$, as higher the value of the quantity $1 - \frac{m_\nu^2}{(Q - E'_j)^2}$ deviates significantly from the unit. This means that the spectral lines undergo a deformation caused by a finite value of $m_\nu$ which will increase the more the value of the quantity $Q - E'_j$ will decrease. The effect of a nonzero neutrino mass, in EC decay, near the end-point, is very similar to the one from $\beta$-decay.

The capture process cannot be detected directly due to the extremely low interaction probability of the emitted neutrino [59]. The capture rate can therefore only be determined from the intensity of subsequently emitted radiation, such as X-rays or Auger electrons, given off during reorganization of the electronic cloud after capture, and $\gamma$-rays or conversion electrons from the daughter nucleus.
3 Microcalorimeters with Transition Edge Sensors

A microcalorimeter records the energy due to a single event (e.g. photon, beta decay). In an ideal model, the microcalorimeter is treated as a single heat capacity device, while a real microcalorimeter consists of many different components connected via finite thermal conductance.

This Chapter will be focused on some important aspects of cryogenic microcalorimeter such as its working principle and its modelling, and also a Transition Edge Sensor used for cryogenic microcalorimetry will be described.

3.1 Microcalorimeter Working Principle

A microcalorimeter detector ideally measures all the energy released in any $\beta$ or EC decay except the energy from the electron neutrino, $\nu_e$. This detector consists of three parts, absorber, sensor (thermometer) and a heat sink, and its performance optimization requires the full characterization of the detector parts. The absorber is the part of the detector that absorbs the energy of the event, such as X-rays from space or from a beta decay, and converts it into heat, thermalizing the energy. The sensor, a thermistor or a TES, is the part that measures the energy rise in the absorber, changing its electrical resistance dramatically with a small change in temperature. The heat sink absorbs the heat from the detector, keeping it cool, in order for the detector return to its initial conditions, and be ready to detect a new event.

The absorber, with the thermal capacity $C$, is in strong thermal contact with the sensor, which in turn, the latter is placed in weak thermal contact with the heat sink, characterized by the thermal conductance $G$, as shown in Fig. 3.1. In the absence of input power, such as a power supply, or energy from the incident particle, other than Joule power, the temperature of the absorber is higher than the temperature of the heat sink due to the Joule power dissipated into the thermometer resistance. If an event occurs, the incident radiation on the detector releases energy in the absorber, causing a local temperature increase. The heat sink allows the system to return to its working temperature after a certain characteristic time (relaxation time). The detector relaxation time, $\tau$, is proportional to the heat capacity, $C$, and inversely proportional to the thermal conductance, $G$, [44] as in Fig. 3.2. The rise time, the time required for the device detect an event, which in this case is the time needed for the temperature of the device reach it's a value from maximum value, assuming that the absorber is 100% efficient, which means that it converts all the deposited energy into thermal energy, must be fast enough to recognize the energy release of different events, to avoid their overlap (pile-up), but long enough to allow all the energy of the event to turn into heat. This time constant depends on the absorber, where the temperature rise upon absorption is proportional to the energy deposited in the absorber and inversely proportional to the heat capacity, as shown in Fig. 3.2.

The configuration of a bolometer is exactly the same, but the only difference is that a bolometer measures the power $P$ deposited on the detector, usually from a flux of incident photons.
The rise time, for HOLMES \( \sim 1 \mu s \) [61], is a characteristic of the absorber material and is generally much smaller than the decay time, few tens of \( \mu s \). The effective time constant \( \tau_{eff} \) of the detector is determined by \( C \), \( G \) and the electrothermal feedback, which will be explained latter in Chapter 5. If an event occurs at \( t=0 \), the temperature variation of the detector is defined as:

\[
\Delta T = \frac{E}{C} e^{-t/\tau_{eff}}
\]

From Eq. 3.1, one can determine the energy and the timing of the incident particle by recording the temperature variation of the detector.

In the design and construction of microcalorimeters that fulfill the requirements of neutrino mass experiments, it is important to optimize the performance of the absorber and the sensor, since they are the most critical parts of a detector. To achieve a measurable temperature change for a small energy deposition, meaning a high temperature variation after irradiation, the absorber should have a high stopping power, and a low heat capacity, \( \sim 10^{-2} J/K/m^3 \) [63]. Also, for a low \( \tau \) value, the system rapidly return to its initial conditions and detect a new event, a low heat capacity value of the system is needed.

A low operating temperature, \( \sim 0.1 K \), is required so that the energy deposited is large relative to the random transfer of heat across the weak link. For negligibly small rise time, \( \sim 100 \mu s \), the conductivity of the absorber must be very high, \( \sim 5 W/K/m \) [63] at the working temperature range, 0.1 K.

At low temperatures, the specific heat of a pure crystal is expressed as:

\[
c(T) = c_r(T) + c_{el}(T)
\]  

(3.2)

where \( c_r \) represents the lattice contribution , due to lattice vibrations, and \( c_{el} \) represents the electronic contribution, due to electronic excitation.

The lattice contribution at very low temperature, \( T \ll \Theta_D \), is given by [56]:

\[
c_r(T) \approx \frac{12\pi^4}{5} k_B N_A (\frac{T}{\Theta_D})^3 = aT^3
\]  

(3.3)

where \( k_B \) is the Boltzmann constant, \( 1.381\times10^{-23} J/K \), \( N_A \) is the Avogadro number, \( 6.022\times10^{23} mol^{-1} \), and \( \Theta_D \) is the Debye temperature of the material.

The electronic contribution at very low temperature, \( T \ll \Theta_F \), is given by [56]:

\[
c_{el}(T) = \frac{1}{2} \pi^2 k_B N_A \frac{T}{\Theta_F} = \gamma T
\]  

(3.4)

where \( \Theta_F \) is the Fermi temperature.

From Eq. 3.4 one can see that the higher the Fermi temperature of the metal, the lower the electronic heat capacity.

The experimental specific heat capacity of metals can be expressed as:

\[
c = aT^3 + \gamma T
\]  

(3.5)
where \( a \) and \( \gamma \) are constants characteristic of the metal.

At the temperature range of interest, which is the working temperature of a TES detector (~0.1 K), the electronic contribution dominates the phonon contribution, as shown in Fig. 3.3. Therefore, one can assume that the specific heat capacity of a metal in the working temperature range of a cryogenic microcalorimeter is given by:

\[
c = \gamma T
\]  

(3.6)

In the case of a superconductor material, the specific heat is different from the superconductor to the normal (metallic) state, as shown in Fig. 3.4. The phonon contribution in such a material is the same, whether the material is in the superconducting or normal state, and is given by Eq. 3.3. On the other hand, the electronic contribution for the specific heat in a superconductor varies with the absolute temperature in the normal and in the superconducting state, as shown in Fig. 3.4. At the critical temperature, \( T_c \), there is a discontinuous change in the specific heat, followed by a decrease proportional to \( T^3 \) below \( T_c \). However, for temperatures at \( T < 0.5T_c \), the electronic specific heat in the superconducting state follows an exponential temperature dependence with an argument proportional to \(-1/T\), being lower than in the normal state, so the electronic contribution in this region is negligibly small. Above \( T_c \), the material is at the normal state and the specific heat drops abruptly. At this point the specific heat is given by Eq. 3.4. From Eq. 3.6, one can see that for pure metals at low temperature the electronic contribution to the specific heat dominate. For a superconductor, however, some of the electrons form Cooper pairs and undergo Bose-Einstein condensation into the ground state, thus being enabled to carry thermal energy.

The electronic specific heat of semiconductors decreases exponentially fast with \( T \) at low temperatures, so this type of material, also like metals, has very low heat capacity, due to the absence of electronic contributions at this temperature range.

An important parameter to take into account in any measurement is the energy resolution of the device used, which in the case of a cryogenic microcalorimeter is given by [65]:

\[
\Delta E_{FWHM} \approx \sqrt{\frac{4k_B T^2 C}{\alpha}}
\]  

(3.7)

where \( k_B \) is the Boltzmann constant, \( T \) is the absorber temperature, \( C \) is the total heat capacity, \( \alpha \) is the sensitivity of the thermometer. The detector energy resolution is proportional to the total heat...
capacity, so for a high detector resolution, which implies a low \( \Delta E \) value, it is required the lowest possible heat capacity value.

To achieve a high detector resolution and efficiency, parameters such as, heat capacity, thermal conductivity, diffusivity and stopping power of the absorber material need to be optimize. Metallic, superconducting, and semiconducting materials have been tested for many decades [16, 17] to be used as the microcalorimeter absorber. The metallic absorbers have relatively large heat capacity, at the working temperature region of a microcalorimeter, but a fast detector response. In many cases the choice of absorber materials is also determined by the choice of thermometers, and, it was shown the possibility of using metallic absorbers with TES based microcalorimeters. Devices with tunable heat capacity and energy resolution of 2.4 eV (gold) and 2.1 eV (gold/bismuth) FWHM at 5.9 keV have been fabricated [63].

3.2 Microcalorimeter Modelling

The simplest thermal model with which a microcalorimeter detector can be schematized is as an absorber of heat capacity \( C \), in thermal contact, through a conductance \( G \) to the heat sink, where the sensor (thermometer) has negligible heat capacity as superconducting, and is at the same temperature as the absorber [66].

The microcalorimeter sensor must be capable of measuring a small variation in the temperature, \( \sim 45 \) mK, for a gold absorber of for example, \( 50 \times 50 \times 5 \) \( \mu \)m\(^3\), and an energy deposition of 2.5 eV, and Transition Edge Sensors (TES) have become very popular thermometers for low-temperature detectors. For this reason in this work I will focus on TES.

The temperature sensitivity of a resistive thermometer is defined through the relation [67]:

\[
\frac{dR}{R} = \alpha \frac{dT}{T} \Rightarrow \alpha = \frac{T}{R} \frac{dR}{dT}
\]  

(3.8)

where \( R \) and \( T \) are respectively the resistance and the temperature of equilibrium of the sensor; for a superconductor, \( \alpha \) is always higher than zero.

The thermal conductance is defined as [66]:

\[
G = \frac{dP}{dT}
\]  

(3.9)

where \( dP \) is the infinitesimal power through the heat link to the heat sink and \( dT \) is the temperature difference between the detector and the heat sink due to that power. In general, \( G \) can be expressed as a power law of the temperature of the type [66]:

\[
G = G_0 T^\beta
\]  

(3.10)

A constant Joule power, \( P_J \), is dissipated into the detector due to the applied small bias current for the thermometer resistance read out of a microcalorimeter. In thermal equilibrium, the power flowing out through the weak thermal link to the heat sink is equal to the power dissipated into the detector. If \( P_J \) is the only input power, the equilibrium state is reached when the total power leaking through the heat link is equal the Joule power. In equilibrium conditions, with no other contribution of power than that dissipated by the Joule effect, necessary to measure the resistance of the thermometer, the equilibrium temperature \( T \) is determined by integrating the Eq. 3.9 [66]:

\[
\int_{T_S}^T G(T')dT' = P(T) = P_J(T)
\]  

(3.11)

where \( T \) is the detector equilibrium temperature and \( T_S \) is the temperature of the heat sink. \( T \) is always higher than \( T_S \).

Substituting Eq. 3.10 in Eq. 3.11, one obtains:
Dimensionally, $G$ and $G_0$ are different but the numerical value of $G_0$ is equal to the value of $G$ at 1K, [66]. It is important to remember that the Joule power is a function of the resistance of the thermometer, and then of the temperature, in other words, the temperature dependence of the Joule power is due to the temperature dependence of the thermometer resistance. To calculate the equilibrium temperature, it is therefore necessary to solve the system composed by Eq. 3.12, $P(R)$ and $R(T)$, and in general it can be solved numerically.

If the detector is kept perfectly isolated from power sources other than the Joule power and now one considers an additional power, $W$, deposited in the detector, it is possible to calculate the resulting temperature rise, $\Delta T$, above the equilibrium. Since a portion of the total power ($P + W$) that reaches the sensor is accumulated in the detector heat capacity $C$ (combined heat capacity of the thermometer and absorber), and another part flows to the heat sink through the thermal link with conductance $G$, the total power input must be equal to the sum of the increase in internal energy of the detector and to the power flowing out through the thermal link. The equation for determining the generic detector temperature, $T_D$, is [66]:

$$C \frac{dT_D}{dt} + \int_{T_s}^{T_D} G(T') dT' = W + P(T_D)$$ (3.13)

where $T_D$, $W$ and $P$ are functions of the time, $t$. The first term in the left side represents the power stored into the heat capacity of the detector, while the second term represents the power leak from the detector (rate of flow of heat from to the heat sink via the thermal link).

If the temperature sensor is expressed as $T_D = T + \Delta T$, Eq. 3.13 can be written as:

$$C \frac{dT + \Delta T}{dt} + \int_T^{T + \Delta T} G(T') dT' = W + P(T + \Delta T)$$ (3.14)

In the small signal limit ($\Delta T \ll T$), the second integral can be expanded using the Taylor expansion formula. Considering only the first order of $\Delta T$, the second integral in Eq. 3.14 becomes:

$$\int_T^{T + \Delta T} G(T') dT' = \int_T^{T + \Delta T} [G(T) + \frac{dG(T')}{dT'}] dT' \approx G(T) \Delta T$$ (3.15)

Using Eq. 2.15 and recalling Eq. 3.11 in Eq. 3.14 one obtains:

$$C \frac{dT + \Delta T}{dt} + P(T) + G(T) \Delta T = W + P(T + \Delta T)$$ (3.16)

Defining $\Delta P = P(\Delta T + T) - P(T)$ and using $G(T) = G$ for simplicity, Eq. 3.16 becomes [66]:

$$C \frac{d\Delta T}{dt} + G \Delta T = W + \Delta P$$ (3.17)
Considering the detector powered by a circuit as in Fig. 3.5, where $R$ is the resistance of the thermometer and $R_L$ is the load resistance, when using a direct current power supply, for which must be the relationship $R_L \gg R$, the detector is said to be constant current biased, for a direct voltage power supply the load resistance needs to have a much lower value than $R$, $R_L \ll R$, and the detector is said to be constant voltage biased for voltage supply.

Using the expression for the dissipated power, $P = I^2R = \frac{V^2}{R}$, and differentiating it with respect to the temperature one gets:

$$\frac{dP(T)}{dT} = \frac{2V}{R} \frac{dV}{dT} - \frac{V^2}{R^2} \frac{dR}{dT} = \frac{2V}{R} \frac{dR}{dT} - \frac{V^2}{R^2} \frac{dR}{dT} = \frac{dR}{dT} \left( \frac{2V}{R} \frac{dV}{dR} - \frac{V^2}{R^2} \right)$$

(3.18)

Replacing Eq. 3.8 in Eq. 3.18, where $dR/dT = \alpha R/T$:

$$\frac{dP(T)}{dT} = \frac{\alpha R}{T} \frac{V^2}{R} \left( \frac{2R}{V} \frac{dV}{dR} - 1 \right) = \frac{\alpha}{T} \frac{2R}{V} \frac{dV}{dR} - 1$$

(3.19)

For the circuit in Fig. 3.5, for which $V = \frac{R}{R+R_L} V_{Bias}$, the derivative of the voltage as a function of the resistance is:

$$\frac{dV}{dR} = \frac{R_L}{(R+R_L)^2} V_{Bias}$$

(3.20)

Substituting Eq. 3.20 in Eq. 3.19 and replacing $V$ by $V_{Bias}$ one gets:

$$\frac{dP(T)}{dT} = \alpha \frac{P}{T} \left( \frac{2R}{R+R_L} \frac{R_L}{R_{Bias}^2 (R+R_L)} V_{Bias} - 1 \right) = -\alpha \frac{P (R-R_L)}{T (R+R_L)}$$

(3.21)

In the case of small signals it can be written [66]:

$$\Delta P = -\alpha \frac{P (R-R_L)}{T (R+R_L)} \Delta T$$

(3.22)

This term is called electrothermal feedback (ETF) and plays a fundamental role in the response of the detector. For simplicity, it can be written as [66]:

$$\Delta P = -G_{ETF} \Delta T$$

(3.23)
where \( G_{ETF} = \frac{P}{C} \left( \frac{R - R_L}{R + R_L} \right) \) is called virtual thermal conductance and the effective thermal conductance is then given by \( G_{eff} = G + G_{ETF} \).

The sign of ETF is determined by the sign of \( \alpha \) and the magnitude of \( R_L \), being positive if the bias power \( P \) increases with the increase in temperature \( T \) and vice versa. A voltage biased thermistor produces positive ETF, while a voltage biased TES produces negative ETF. Similarly, a current biased thermistor produces negative ETF and a current biased TES thermometer produces positive ETF [44].

In this way Eq. 3.17 can be written as:

\[
C \frac{d(\Delta T)}{dt} + G \Delta T = W - G_{ETF} \Delta T \Leftrightarrow C \frac{d(\Delta T)}{dt} + G_{eff} \Delta T = W \tag{3.24}
\]

The increase in temperature caused by an energy \( E \) incident on the absorber is of the type:

\[
\Delta T(t) = \frac{E}{C} e^{-t/\tau_{eff}} \tag{3.25}
\]

where \( \tau_{eff} = \frac{C}{G_{eff}} = \frac{C}{G + G_{ETF}} \) is the time to thermalization. Eq. 3.25 is the response of the microcalorimeter or bolometer, the time domain solution, assuming a negligible rise time compared to \( \tau_{eff} \), calculated using Eq. 3.24, since for a single event with energy \( E \) absorbed by the detector at time \( t_0 = 0 \), \( W \) is a delta function of the form \( W = E \delta(t - t_0) = E \delta(t) \). After absorption of a single event, the temperature of the detector decays exponentially with the time constant \( \tau_{eff} \).

Let us consider both current bias and voltage bias conditions to see the effect of the ETF on the performance of the microcalorimeter.

Under current bias condition where \( R_L \gg R \), meaning a positive electrothermal feedback one has (see Eq. 3.23):

\[
G_{ETF} = -\frac{aP}{T} \text{ and } \tau_{eff} = \frac{T}{1 - \frac{aP}{GT}} \tag{3.26}
\]

where \( \tau = C/G \), is the intrinsic time constant regardless the feedback.

Furthermore, for a voltage bias condition where \( R_L \ll R \), under negative electrothermal feedback one has:

\[
G_{ETF} = \frac{aP}{T} \text{ and } \tau_{eff} = \frac{\tau}{1 + \frac{aP}{GT}} \tag{3.27}
\]

For example, in a TES, the material used is superconducting, where the parameter \( \alpha \) is always higher than zero, and so the quantity \( aP/GT \) is positive. In this type of detector, for a positive ETF the effective time constant, \( \tau_{eff} \), is higher than the intrinsic time constant, \( \tau \), so the detector takes a longer time to return to its initial conditions. In a TES under negative ETF, the effective time constant is smaller than the intrinsic time constant. Thus, the negative ETF reduces the decay time (pulse duration), making the detector faster.

Moreover, considering the stability condition of the system, namely that the argument of Eq. 3.25 is negative, one has:

\[
\tau_{eff} > 0 \Rightarrow \frac{C}{G + G_{ETF}} > 0 \Rightarrow G_{ETF} > -\frac{G}{GT} \Rightarrow \frac{aP}{GT} > -1 \tag{3.28}
\]

This condition is always verified in the case of a TES under negative electrothermal feedback, since in this case \( G_{ETF} \) is always higher than zero. In a detector, under this condition, the pulse energy is dissipated by the Joule effect, rather than an actual transport of the heat to the heat sink, making a voltage biased detector stable against the thermal runaway. A decrease in the joule power with an increase in the temperature acts as a restoring force. Therefore, being the detector power
input the Joule power $V^2/R$ and the power output to the heat sink through the weak thermal link given by Eq. 3.12, in thermal equilibrium, one has:

$$P = \frac{V^2}{R} = \frac{G_0}{\beta + 1} (T^{1+\beta} - T_s^{1+\beta})$$

(3.29)

Replacing Eq. 3.10 in Eq. 3.29, the power can be written as:

$$P = \frac{G_0}{\beta + 1} (T - \frac{T_s^{1+\beta}}{T})$$

(3.30)

To obtain the term $aP/GT$ from Eq. 3.30 one multiplies both sides of the equation by $1/T$ and $a$, getting:

$$\frac{aP}{GT} = \frac{a}{\beta + 1} \left[ 1 - \left( \frac{T_s}{T} \right)^{\beta + 1} \right]$$

(3.31)

Substituting Eq. 3.31 in Eq. 3.27 for $\tau_{eff}$ one obtains:

$$\tau_{eff} = \frac{\tau}{1+\frac{\alpha}{\beta + 1} \left[ 1 - \left( \frac{T_s}{T} \right)^{\beta + 1} \right]} = \frac{\tau}{1+\frac{\alpha}{\beta + 1}}$$

(3.32)

where $\Phi = 1 - \left( \frac{T_s}{T} \right)^{\beta + 1}$ and its value for a TES detector varies between 1 and 0 depending on the temperature of the superconducting film and the heat sink.

In the case of "extreme" negative electrothermal feedback, that is, when $T \gg T_s$ and $\frac{\alpha}{1+\beta} \gg 1$, the thermalization time becomes:

$$\tau_{eff} = \frac{\tau}{1+\frac{\alpha}{\beta + 1}} \approx \frac{\tau(\beta + 1)}{\alpha}$$

(3.33)

In principle it is possible to decrease the time constant by increasing the value of $G$, but the input power must be increased proportionally to keep the thermometer in the working temperature range. Practical limitations come from the fact that input power cannot be increased above a certain boundary to avoid nonlinear effects. Therefore after a limit, the only way to decrease the time constant of the detector is by increasing $\alpha$.

The condition where the energy is dissipated by Joule effect, allows one to express the incident energy $E_{\chi}^{\text{meas}}$ as [65]:

$$E_{\chi}^{\text{meas}} = \int_{0}^{\infty} |\Delta P_{\text{joule}}| dt = V \int_{0}^{\infty} |\Delta I| dt$$

(3.34)

where a considerable simplification in the analysis of the pulse is made. In fact, non-uniformity of the film and non-linearity in its transition curve $R(T)$ can affect the shape of the pulse, but not its time integral.

Introducing the thermal efficiency of the detector, defined in general as the ratio between the energy measured, $E_{\chi}^{\text{meas}}$, and the actual energy deposited in the absorber, $E_{\chi}$:

$$\epsilon_{th} = \frac{E_{\chi}^{\text{meas}}}{E_{\chi}}$$

(3.35)

One possibility to improve the thermal efficiency is to build detectors suspended above a thin layer (about 1 micron) of silicon nitride, which greatly reduces the escape of phonons. In the case where the system satisfy the conditions for an "extreme" electrothermal feedback, $G_{\text{EFF}}$ is
dominant with respect to \( G \), so increasing the value of \( G \) has little influence on the thermalization time.

The quantity \( \Delta I \) which appears in Eq. 3.25 is the change in current due to the change in resistance of the detector, and is the physical quantity that is actually measured. Its dependence on temperature variation, in the case of the circuit in Fig. 3.5, is obtained by differentiating the expression \( I = \frac{V}{R} \) in a manner similar to that done for the power dissipated as a function of temperature, as in Eq. 3.18, namely [66]:

\[
\frac{dI}{dT} = \frac{1}{R} \frac{dV}{dT} - \frac{V}{R^2} \frac{dR}{dT} = \frac{dR}{dT} \frac{V}{R^2} - \frac{V}{R^2} \frac{dR}{dT} = \frac{-\alpha l}{R+R_L} \frac{I}{R} \frac{R}{T} \frac{1}{R+R_L} \approx -\alpha \frac{l}{R} \frac{R}{R+R_L} \Delta T
\]  

(3.36)

and in the case of small signals can be written:

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

(3.37)

It is clearly seen from Eq. 3.28 that an increase in temperature corresponds to a decrease of the measured current, a consequence of the fact that also the resistance of the detector increases.

For a voltage bias detector, \( R_L \ll R \), and so Eq. 3.28 is reduced to:

\[
\Delta I \approx \alpha \frac{l}{R} \Delta T = -\alpha \frac{l}{R} \Delta T = -\alpha \frac{V}{TR} \Delta T
\]  

(3.38)

Substituting Eq. 3.25 in Eq. 3.29 one obtains:

\[
\Delta I = -\alpha \frac{V E_x}{TR} e^{-t/\tau_{eff}} = \Delta I_0 e^{-t/\tau_{eff}}
\]  

(3.39)

where \( \Delta I_0 = -\alpha \frac{V E_x}{TR} \) is the initial drop in current due to the absorption of energy \( E \).

### 3.3 Transition Edge Sensor Detector

The Transition Edge Sensor, TES, is a superconducting thin film, maintained at a temperature in the transition region of superconducting to normal states. It operates in the Transition Phase. In this detector, the incident radiation causes a partial transition of the film, due to a temperature rise, which causes a change of the thermometer electrical resistance. This type of detector is used in the linear region of the transition [65], as shown in Fig. 3.6. In this regime, the change in resistance is proportional to the change in temperature, and therefore to the energy deposited in the absorber. Through the characterization of the \( R(T) \) curves is possible to obtain the temperature rise and, knowing the thermal capacity of the system (absorber plus thermometer) is possible to calculate the energy transferred to the absorber by the incident radiation using Eq. 3.25.

The typical working temperature range for a TES detector is between 30 mK up to 1 K depending on the superconductor material and the TES designs and applications [67, 68, 69]. For MARE and HOLMES experiment the working temperature is \( \sim 100 \) mK [70], which is much below both Debye and Fermi temperatures and its thermal efficiency, \( \varepsilon_{th} \), for a TES that uses a silicon substrate, is around 50% [71].
The major complexity in using TES is to stabilize it within the narrow transition temperature range, ~ some mK, which is fixed by the manufacturing process. A tiny variation in temperature leads the material to the superconductor or normal states, decreasing the detector sensitivity. The Joule heating can cause thermal drift and temperature fluctuations that, even if small, can significantly degrade its performance. The shape of the transition itself is heavily influenced by the geometry of the sensor and from edge effects, such as imperfections in the contours and inside the film. Normally, the TES superconducting film is made of iridium, due to its sharp transition in a temperature range between 0.118 and 0.122 K depending on the bias current, as showed by a study done at the Miami University [72].

When a TES is biased with a current source, \( R_L \gg R \), the Joule heating, \( P(T) = I^2 R(T) \), increases with increasing temperature and the ETF is positive. On the other hand, if the TES is biased with a voltage source, \( R_L \ll R \), the Joule heating, \( P(T) = V^2 / R(T) \), decreases with increasing temperature and the ETF is negative. In this way, the negative ETF stabilizes the close loop gain making circuit performance uniform and reproducible. It also reduces nonlinearity effects and increases the dynamic range. Therefore, TES microcalorimeters are commonly voltage biased, which counteracts deviations from the set point, and therefore these detectors can be self-biased and self-calibrated within the transition, over a certain range of signal power and biased voltage [73]. If the heat sink is well below the transition temperature, the TES response is almost insensitive to the temperature fluctuation in the sink and hence the TES temperature is stabilized.

These calorimeters allow to achieve excellent energy resolutions and another important feature is the rapid response time, due to the negative electrothermal feedback. For \( \alpha \leq 100 \), the time constant of a TES detector, \( \tau_{eff} \), as shown in Eq. 3.33, is shortened by more than two orders of magnitude with negative ETF [74]. Fast response does not improve the signal to noise ratio, however, it does increase the useful count rate and avoids the possible degradation in energy resolution due to the pulses piling up. Transition edge sensors have the highest known \( \alpha \) and therefore are becoming the detector of choice [44].

The application of voltage biased operation with a superconducting quantum interface device (SQUID) has led to a rapid growth in the development and application of TES detectors. SQUIDs can be easily impedance-matched to low-resistance TES detectors, few Ohms, and it is also possible to multiplex the readout of TES detectors using SQUID amplifiers [75]. Arrays of large
number of TES detectors are now being investigated and developed for a number of different applications [76, 77].

The structure of a typical TES detector is shown in Fig. 3.7. This detector type is being tested for MARE and HOLMES experiments and it will consist of Ir-based superconducting films deposited on Si-substrates. The substrate, on which is built the TES, is a silicon (100) wafer of 5×5×1 mm³. On the Si substrate was deposited, by the manufacturer, a silicon nitride (Si₃N₄) layer with a thickness of 1 μm. The center of the silicon wafer was removed, leaving only the silicon nitride membrane with a surface of 250×250 μm². The absorber can be a rhenium crystal with 200 – 300 μg, in the case of MARE experiment or can be a Au, Cu or Bi metal with a surface area of 100×100 μm² and a thickness between 2 – 3 μm, implanted with 1011-1013 nuclei of 163Ho, depending on the activity requirements, in the case of HOLMES experiment [13].

The superconducting sensor, consisting on the layers of Ir and Au, for MARE, and of Mo with Au, Cu or Bi for HOLMES, under the absorber, as shown in Fig. 3.7, have a critical temperature which depends on the thickness [78], and on the proximity effect [79], since a bilayer of a thin superconducting film and a thin normal metal act as a single superconductor with a tunable Tc. Typically, the sensor bilayers can have critical temperatures critical values between 50 and 100 mK [80].

![Fig. 3.7: Thermal model of a real TES detector; C_L and C_el are, respectively, the heat capacity of the phonon and electronic systems [42].](image)

For a more realistic schematic of the heat flow, the TES detector can be divided into several thermally homogeneous systems, in communication via thermal conductances.

In the sensor, at working temperature between 80 and 90 mK, the thermal coupling electron-phonon interaction is weaker than the electron-electron and phonon-phonon interactions. This behavior is typical of metal materials and is described by the Hot Electrons Model [81, 82]. The electrical resistance of the sensor depends on the temperature of its electronic system. Under equilibrium conditions, the electronic system will have a different temperature from the heat sink due to the Joule power. For this reason, normally the temperature of the electronic system is higher than that associated with phonons, and therefore, iridium film can be modeled as a set of two different systems, the electronic and the phonon, joined together by a thermal conductance G_{el}^{L} [42].

The absorber is attached to the thermometer by means of an epoxy resin: their thermal contact is through a conductance G_α, which normally has a high value. Also, in the absorber, electrons and phonons are decoupled. Is the absorber that will absorb the photons with a maximum energy of 2 keV that are intended to detect. The electronic system of the absorber will be excited by photoelectrons, and through their interaction will absorb their energy. It is important to study how
the heat stored in the electronic system of the absorber will propagate inside the detector. A diagram of the thermal coupling is shown in Fig. 3.8. For simplicity the silicon nitride layer will be indicated as SiN.

![Diagram of thermal coupling](image)

**Fig. 3.8**: Thermal model of a real TES detector; $C_{el}$ and $C_i$ are, respectively, the heat capacity of the electronic and phonon systems [42].

The resin between the absorber and the sensor is an amorphous insulator and has a low thermal conductivity. The heat transfer to its interior can be described by a conductance proportional to $T^2$. When the heat reaches the iridium layer, part of the energy is transferred to phonons of the SiN layer, which are then transported to the heat sink, consisting of the silicon layer, through lattice excitation of the lattice. This happens due to the fact that SiN is an insulating material at working temperature allowing only the phonon transport to the heat sink. The other part of the energy is stored in the electrons of iridium.

In the absorber, the heat transfer from the electronic system to the phonon occurs with a thermal conductance such as [42]:

$$G_{e\rightarrow i}^{abs} \propto (T_e^\beta - T_i^\beta)$$

(3.40)

where $\beta$ may vary between 4 and 5, according to the degree of purity and to the ordered structure of the material.

The phonon systems of different materials in contact interact thermally through the Kapitza relative conductance. The thermal conductance in the interface iridium-silicon nitride is described by [42]:

$$G_{i\rightarrow SiN}^{Ir} \propto S(T_{ir}^3 - T_{SiN}^3)$$

(3.41)

where $S$ is the contact surface between the two materials ($\approx 10^4 \mu m^2$).

Similarly, one can write a similar relationship between the absorber and the sensor, as there is a double Kapitza interface in series with the thermal resistance of the resin with a total contribution of $G_a$ as in Fig. 3.8.

Across the sensor there are two electrical contacts in aluminum that allow to power the detector. At working temperature, aluminum is fully superconducting, while iridium is located on the transition region and therefore is still resistive. Anyway, the passage of electrons through the interface metal-superconductor is prevented by Andreev reflection phenomenon [83].

The thermal conductance between iridium and aluminum is given by [42]:

---

**Note**: The exact equations and expressions may vary depending on the specific context and the level of detail required. The text provides a general overview of the thermal coupling and conductance, incorporating key concepts from the referenced literature. The images and diagrams are intended to complement the textual description.
\[ G_{l-1}^{Ir-Al} \propto S'(T_{Ir}^3 - T_{Al}^3) \] (3.42)

where \( S' \) is much smaller than \( S (\approx 20 \, \mu m^2) \), and so the heat flow between iridium and aluminum can be neglected.

As shown in Fig. 3.7, the detector is constructed above a suspended membrane of silicon nitride (SiN), whose function is to slow down the thermalization process. The thermal conductance of this membrane phonon system, \( G_{l-1}^{SiN} \) is in fact very small. Some studies [84] have considered the transfer of heat along a SiN membrane similar to the case of the radioactive heat exchange between two black bodies. In this case, the thermal conductance of silicon nitride membrane is described as:

\[ G_{l-1}^{SiN} = 4\sigma A T^3 \xi \] (3.43)

where \( A \) is the cross section perpendicular to the flow of heat and \( \sigma \), equal to 15.7 mW/(cm²K), is the Stefan-Boltzmann constant, obtained by summing the transverse and longitudinal modes of the propagation of phonons and using their sound speed in SiN membranes. The parameter \( \xi \) allows to fix an upper and lower limit for the value of \( G_{l-1}^{SiN} \), describing the strong dependence of the heat transfer in the diffusion of phonons on the membrane surface. In the case of \( \xi = 1 \) there is specular surface scattering, and this condition gives the maximum value for \( G_{l-1}^{SiN} \). If \( \xi < 1 \) there is a reduction in the conductance due to the diffusive surface scattering. The minimum value for \( G_{l-1}^{SiN} \) is in the case of completely diffusive surface scattering, called Casimir limit. Since the transmission of heat through the membrane is strongly conditioned by the diffusive scattering, the conductance in the membrane has very low values. This membrane has a very important role in the detection mechanism, since \( G_{l-1}^{SiN} \) limits the heat flow to the heat sink, allowing the electronic system of iridium to warm up, and to be able of measuring the resulting change in electrical resistance.

In summary, for the thermal model of the TES, the following conditions must be fulfilled [42, 85]:

\[ G_{e-1}^{Ir} \gg \left( \frac{1}{G_{l-1}^{SiN}} + \frac{1}{G_{l-1}^{Ir-SiN}} + \frac{1}{G_{l-1}^{SiN-Si}} \right) \rightarrow G_{e-1}^{Ir} \gg G_{l-1}^{SiN} \] (3.44)

Further reduction of the heat flow to the heat sink is achieved by adding metallic impurities within the membrane, or by drilling holes that modify the geometry, thus conditioning the phonon trajectory.

The detailed processes through which the radiation dissipates the energy in the detector are not fully known, so it is assumed a ionization mechanism where radiation produces electron-hole pairs, which in turn produce more pairs according to two principal ways of energy transfer: creation of pairs with an expenditure of energy \( E_i \) and the generation of phonons with an energy \( \Omega \) by charge carriers with an energy \( E > E_i \). At the end of the process each charge will have a kinetic energy \( E_f \) lower than \( E_i \) [42, 85].

The achievement of high efficiency in the thermalization process is a necessary condition for approaching the performance limit of the intrinsic calorimeter. In metals the heat conversion process is faster than in semiconductors, since there is no band gap and so the de-excitation of the electronic states is more favored. The thermal equilibrium can be reached within the time of formation of the signal and the efficiencies are close to 1. For this reason, metals are the main choice for absorbers which have been achieved eV resolutions FWHM at 6 keV.

In this work, a gold absorber is considered to study the process of holmium implantation for HOLMES experiment.
4 Microcalorimetric Measurements of the $\beta$-decay for neutrino mass direct determination

Several experiments are trying to determine the absolute mass value of the electron neutrino. The strongest upper limit on the neutrino mass comes from cosmological data such as cosmic microwave background radiation, galaxy surveys and Lyman-alpha forest indicating that the sum of the neutrino masses must be less than 0.3 eV [86].

Direct measurements of neutrino mass are carried out by analyzing the kinematics of charged particles emitted together with flavor state neutrinos. To date the most sensitive electron neutrino mass measurement is based on studying the deformation of the beta spectrum close to the end-point energy [87]. Experiments that rely only on the energy momentum conservation in the beta decay are the only model independent method to determine the electron neutrino mass with a sub-eV sensitivity.

To date, the most sensitive limits for electron neutrino mass have been achieved using spectroscopic measurement of tritium beta decay. Tritium beta decay is an allowed transition given by:

$$^3H \rightarrow ^3He + e^- + \bar{\nu}_e$$

(4.1)

where $^3H$ is tritium, $^3He$ is helium-3, $e^-$ is the beta particle, the electron and $\bar{\nu}_e$ is the electron antineutrino.

This transition has low end-point energy $E_0 = 18.6$ keV and a short half-life of $T_{1/2} = 12.3$ years. Due to the very high intrinsic decay rate of $^3H$, it is possible to use thin sources, and the high activity is very suitable for an external source using a high resolution electrostatic spectrometer.

A summary of the four most sensitive experiments using spectrometers is presented in Table 4.1.

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Type</th>
<th>Source</th>
<th>$m_\nu$ (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LANL [88]</td>
<td>M</td>
<td>T$_2$ (gas)</td>
<td>&lt; 9.3</td>
</tr>
<tr>
<td>LNLN [89]</td>
<td>M</td>
<td>T$_2$ (gas)</td>
<td>&lt; 7</td>
</tr>
<tr>
<td>Troitsk [90]</td>
<td>E</td>
<td>T$_2$ (gas)</td>
<td>&lt; 2.05</td>
</tr>
<tr>
<td>Mainz [91]</td>
<td>E</td>
<td>T$_2$ (solid)</td>
<td>&lt; 2.3</td>
</tr>
</tbody>
</table>

Table 4.1: Four most sensitive results obtained from spectrometric measurements; M stands for magnetic spectrometer and E stands for the electrostatic spectrometers [44].

From Table 4.1, the most precise experimental results are obtained from the Mainz and Troitsk experiments. The Mainz group [91] used a frozen molecular tritium source whereas the Troitsk [92] utilized a gaseous molecular tritium source. Both groups used similar electrostatic spectrometers to measure the beta spectrum.

There are two main advantages that have made spectrometers the most sensitive devices: only electrons with energies very close to the end-point energy are chosen and, in this way, the required statistics in the region of interest can be obtained in a reasonable amount of time; achievement of very high energy resolution.

The major disadvantages of the spectrometer come from the fact that the radioactive source is external to the detector. In particular, the experimental data are affected by the energy loss due to excitation of atoms or molecules in the source, backscattering from the detector, and reflection from the source substrate.

A larger expansion on the MAINZ and TROITSK experiments, named the Karlsruhe Tritium Neutrino (KATRIN) experiment [93], currently in development, will also use an electrostatic spectrometer. The main spectrometer will analyze the high-energy part of the $\beta$-decay electrons which carry information on the neutrino mass with an energy resolution of $E = 0.93$ eV at 18.6 keV [94].
An alternative to spectrometric measurement of the $\beta$-spectrum is the microcalorimetric approach, where the $\beta$-source is embedded in the detector and all the decay energy is measured, except the one from the neutrino. Despite the fact that the most stringent results come from spectrometers, both the calorimetric and spectrometric methods are two complementary techniques with the same goal, due to the different systematic effects of both techniques. The large weight of systematics in the spectrometric method, requires that confidence in the results can be obtained only through confirmation by independent experiments. The best option is then the calorimetric one, which is characterized by different systematics.

Since, in a calorimetric experiment the source is internal to the detector, so this type of measurement is free from the systematic uncertainties of external sources. The main disadvantage of a calorimetric approach comes from the fact that the entire beta spectrum is recorded. Consequently, one must keep a low count rate, i.e. a low source activity, to avoid pulse pile-up effect, in order to keep a spectral shape without distortions. This leads to a serious limitation on the accumulation of statistics. This limitation can be partially overcome by choosing a $\beta$-source with the lowest possible end point energy, and limited source activity.

To date $^{187}$Re and $^{163}$Ho are the only possible $\beta$-sources for calorimetric measurement, since their decay have the lowest known Q-value.

4.1 $\beta$-DECAY OF $^{187}$Re ISOTOPE

The experimental technique, which is sensitive to the absolute mass of the electron antineutrino, in a model-independent way, is the $\beta$-decay endpoint measurement.

The $^{187}$Re beta decay is given by:

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

where $^{187}$Re is rhenium isotope, $^{187}$Os is the osmium isotope, $e^-$ is the electron, and $\bar{\nu}_e$ is the electron anti-neutrino.

This element has a transition energy of $Q = 2470 \pm 1$(stat) $\pm 4$(syst) eV, [16] the lowest known in nature, and the half lifetime, composable to the age of the Universe, of $4.12 \pm 0.02$(stat) $\pm 0.11$(syst) $\times 10^{18}$ years [16]. The large isotopic abundance (62.8%) of $^{187}$Re in natural rhenium allows getting a useful source without any isotopic separation process. The beta decay rate in natural rhenium is of the order of 1 Bq/mg (one decay per second per milligram). At temperatures below 1.6 K, metallic rhenium becomes superconducting and therefore, possesses very low heat capacity at working temperature of a microcalorimeter. All these characteristics, make $^{187}$Re one of the most suitable elements for a calorimetric measurement.

As mentioned, in a calorimetric experiment the energy detected is the electron energy plus the energy of the excited atomic state and there is only one end point energy, equal to the total energy available in the nuclear decay. However, due to the fact that in this type of experiment all the decays are detected, with energies close or far away from the endpoint, there is a need of huge amount of data to be acquired. $^{187}$Re is a good candidate since it has the lowest known energy endpoint, which means it is the isotope with the highest ratio between events near endpoint and total events.

So far, the MANU experiment at Genoa (Italy) [16] and the MIBETA experiment at Milano (Italy) [4] are the only two experiments based on calorimetric measurement of rhenium beta decay.

The MANU experiment used a 1.5 mg metallic Re single crystal attached with a neutron transmutation doped (NTD) thermistor and collected a statistic of about $10^7$ decays at a temperature of 60 mK. This experiment yielded an endpoint energy of $E_0 = 2470$ eV and an electron neutrino mass upper limit of $m_{\nu_e} < 19$ eV at 90% C.L [13].

The MIBETA experiment, conducted by the collaboration of the Milano and the Como group, showed that the result obtained from a single detector system can be significantly improved by applying an array of microcalorimeters.
The MIBETA experiment used an array of eight microcalorimeters. Each calorimeter was equipped with an absorber containing a mono-crystal of silver perrhenate (AgReO$_4$) with a mass of 300 μg and a silicon implanted thermistor. Fig. 4.1 shows the Kurie plot of Re β-spectrum obtained from the high statistics calorimetric measurements, $6.2 \times 10^6$ counts obtained from 5000 hrs running.

![Fig. 4.1: The Kurie plot for the Re β- spectrum obtained from eight detectors used in the MIBETA experiment (left); the fit residuals and fit function overlaid to the experimental data in the end-point region (right) [4].](image)

In Fig. 4.1, $p$ is the electron momentum, $E$ is the electron kinetic energy, $F(Z, E)$ is the Coulomb factor and $S(E)$ is the shape factor.

The upper limit for electron neutrino mass obtained from the MIBETA experiment is $m_{\nu_e} < 15$ eV at 90% C.L.

### 4.2 The MARE Project

The electron energy spectrum is reaches a maximum value determined by energy difference of the nuclear initial and final states and the mass of the electron anti-neutrino according to Eq. 2.42.

The “Microcalorimeter Arrays for a Rhenium Experiment” (MARE) is an experiment which aims to determine the mass of the electron neutrino, using the calorimetric measurement technique for $^{187}$Re beta decay. This is a very sensitive process due to the very low energy available to the particles in the final state $Q = 2.47$ keV, using a full kinematical approach. The neutrino mass will be determined from the shape of the beta spectrum in the region close to the end point $Q$.

In a calorimetric detection the $^{187}$Re source is embedded in the detector and only the neutrino energy will escape the direct detection. The energy of the neutrino is then measured in the form of the missing energy of the beta decay.

The MARE experiment, is an international project, divided in two stages. The first stage, named MARE 1, had the goal of constraining the neutrino mass up to 2 eV sensitivity, for an energy resolution of $\sim 20$ eV, a rise time of $\sim 100 - 500$ μs [95], where the present sensitivity limits of neutrino mass were achieved using the microcalorimeter technology, and improved. This included the construction of the most sensitive microcalorimeters, such as transition edge sensor (TES) microcalorimeter or metallic magnetic calorimeter (MMC), with the source, $^{187}$Re, embedded in the absorbers. The second stage, MARE 2, is presently being pursued, with the aim of increasing the sensitivity on electron neutrino mass to $\sim 0.1$eV/$c^2$. This last phase is expected to provide a competitive alternative and an independent measure to the results from the KATRIN experiment.
Currently, MARE collaboration is considering both $^{187}$Re and $^{163}$Ho isotopes. The long decay time of the $^{187}$Re isotope puts serious limitations on the design and fabrication of the experiment in MARE 2 phase. The potential of a rhenium $\beta$-decay has already been demonstrated however, due to these limitations, the potential of a holmium electron capture (EC) decay is under course. A parallel experiment to MARE was created, called HOLMES, with the same aim and technologies, but now using $^{163}$Ho instead of $^{187}$Re, which will be explained in chapters 4.3 and 4.4. A comparison between parameter values needed for $^{187}$Re and $^{163}$Ho in MARE/HOLMES experiments are shown in Table 4.2.

<table>
<thead>
<tr>
<th>Isotope</th>
<th>Sensitivity on $m_\nu$ (N.C. 90%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{187}$Re</td>
<td>0.2 eV: 8 arrays (5k pixels each)</td>
</tr>
<tr>
<td>$^{163}$Ho</td>
<td>0.2 eV: 3 arrays (500 pixels each)</td>
</tr>
</tbody>
</table>

$t_{\text{measure}} = 8 \text{ yr}$  $t_{\text{measure}} = 10 \text{ yr}$  $t_{\text{measure}} = 1 \text{ yr}$  $t_{\text{measure}} = 10 \text{ yr}$

Table 4.2: $^{187}$Re and $^{163}$Ho parameters comparison values for MARE/HOLMES experiments [96].

From Table 4.2, one sees that the use of $^{163}$Ho instead of $^{187}$Re allows a higher counting rate per detector.

4.3 EC-decay of $^{163}$Ho Isotope

The region most affected by the effect of $m_\nu \neq 0$ is close to the Q-value of the reaction, as shown in Fig. 4.2, and so this threshold plays a key role. The lower the Q-value, the better the sensitivity in the area of the spectrum more subject to the deformation produced by $m_\nu \neq 0$. It is important to test the best candidates for a direct neutrino mass experiment, and so besides $^{187}$Re, $^{163}$Ho has a Q-value among the lowest, $2.2 < Q_{\text{EC}} < 2.8$ keV [7], with a recommended currently value of $Q_{\text{EC}} = 2.555 \pm 0.016$ keV [97]. Apart from the Q-value, which is approximately the same as $^{187}$Re, a great advantage on using $^{163}$Ho EC decay is its much shorter half-life of 4500 yrs.

$^{163}$Ho decays by electron capture and the total energy emitted as a result of this process roughly corresponds to the binding energy of the electron caught by the daughter atom. This energy is expelled through the emission of X-rays, Auger electrons, Coster-Kronig transitions, and photons emitted by inner Bremsstrahlung.

As already discussed previously, the EC decay spectrum presents a series of peaks, each of which with energy, $E_c = Q - E_\nu$, where $E_c$ is the energy of the c-th peak, i.e. the dissipated binding energy of the electron hole in the final atom.

The process of decay for this isotope is the following:

$$e^- + ^{163}\text{Ho} \rightarrow \nu_e + ^{163}\text{Dy}^c \rightarrow ^{163}\text{Dy} + E_c$$  \hspace{1cm} (4.3)

where $e^-$ is the electron, $^{163}$Ho is the holmium isotope, $\nu_e$ is the electron neutrino, $^{163}\text{Dy}^c$ is the dysprosium excited isotope, and $E_c$ is the de-excitation energy.

This is an allowed transition in which the spin and parity of the nuclei obey the selection rules $\Delta I = 1, \pi_f \pi_j = +1$ [55].
The electron capture by a Ho nucleus provides as product a neutrino, with an energy $E_\nu$, and an dysprosium excited atom, while it presents a gap in one of its innermost shell and one electron a valence shell. It follows that dysprosium, in his excited state, $^{163}$Dy$^\ast$, will naturally decay into its ground state, $^{163}$Dy, emitting the excess energy $E_\circ$.

The electrons that can be captured must obey to the selection rules. The spectrum resulting from the decay for EC will therefore consists of a series of lines, typical of the ionization energies of the electrons captured belonging to the dysprosium atom, and so each will represent the electron capture from a specific shell. The theoretical spectrum of this decay is shown in Fig. 4.3.
These lines have natural width of a few eV, and therefore the real energy spectrum of neutrinos in EC decay is a continuous curve with noticeable peaks, as shown in Fig. 4.3.

As one can conclude from Eq. 4.3, in $^{163}$Ho decay, the end-point energy, determined from the mass difference of the mother and daughter atoms in their ground state, is given by $Q = M(^{163}{\text{Ho}}) - M(^{163}{\text{Dy}})$. The Q-value of this reaction, reaching a value between 2.2 and 2.8 keV makes the only possible electronic levels from which an electron can decay to be $M_1$, $M_2$, $N_1$, $N_2$, $O_1$, $O_2$, as shown in Fig. 4.3. Therefore, only electron from M or higher shells can be captured.

Numerical calculation of Eq. 2.53 can be performed using the $^{163}$Dy atomic parameters, $E_j^*$, which is the ionization energies of the electrons captured in the nucleus of dysprosium from shells $M_1$, $M_2$, $N_1$, $N_2$, $O_1$, $O_2$, $\Gamma_j$ and $\beta_j^2$, given in Table 4.3 [18, 7]. The levels of electrons that can be potentially captured are fully occupied and therefore each $n_j$ is chosen to be 1. The exchange and overlap corrections are neglected by choosing $B_j \sim 1$.

<table>
<thead>
<tr>
<th>Level</th>
<th>$E_j^*$ [eV]</th>
<th>$\Gamma_j$ [eV]</th>
<th>$\beta_j^2 / \beta_{M_1}^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1</td>
<td>2047</td>
<td>13.2</td>
<td>1</td>
</tr>
<tr>
<td>M2</td>
<td>1842</td>
<td>6.0</td>
<td>0.0526</td>
</tr>
<tr>
<td>N1</td>
<td>414.2</td>
<td>5.4</td>
<td>0.2329</td>
</tr>
<tr>
<td>N2</td>
<td>333.5</td>
<td>5.3</td>
<td>0.0119</td>
</tr>
<tr>
<td>O1</td>
<td>49.9</td>
<td>3.0</td>
<td>0.0345</td>
</tr>
<tr>
<td>O2</td>
<td>26.3</td>
<td>3.0</td>
<td>0.0015</td>
</tr>
</tbody>
</table>

Table 4.3: Values of the energy levels of the captured electrons $E_j$, their widths $\Gamma_j$, and electrons squared wave functions at the origin $\beta_j^2$ relative to $\beta_{M_1}^2$ [18, 7].

The heights of the peaks in the energy spectrum of Fig. 4.3 are weakly dependent on the neutrino mass. In general, an EC decay count rate is poor near the end-point, although in the case of Holmium isotope, the count rate in that region is highly enhanced by the M peaks [18].

The results from the Genoa group and from ECHO project, on the calorimetric absorption measurements of $^{163}$Ho, are shown in Fig. 4.4.

The Genoa group used a cryogenic microcalorimeter and sandwiched source for the measurement, and as shown on the left of Fig. 4.4, the group was able to resolve NI, NII, MI and MII...
shells spectral lines that were not previously resolved by other solid state detectors. As for the ECHO project [99], a Ho implanted gold film and MMCs (Magnetic Metallic Calorimeters) were used. The right side of the Fig. 4.4 represents the test results from this project, where the unlabeled peaks in the figure are due to an impurity $^{144}$Pm.

4.4 The Holmes Project

At least three independent methods, the absolute M capture rate or M/N capture rate, Inner Bremsstrahlung (IB), and total calorimetric absorption spectrum end-point, have been suggested to evaluate electron neutrino mass from the Holmium EC decay [7].

The calorimetric method has been considered by embedding the $^{163}$Ho source into the absorber of a microcalorimeter. This experiment was first proposed by DeRujula and Lusignoli in 1982 [58] and it is currently being developed [7]. The capture (calorimetric) energy EC encloses the total de-excitation energy that is detected in the form of electrons and x-rays emitted during the process.

The HOLmium EC MEasurement for electron neutrino mass measurement (HOLMES) aims to determine directly the electron neutrino mass on a pure kinematical basis with sub-eV sensitivity, using arrays of cryogenic microcalorimeters to measure the electron capture decay of $^{163}$Ho. HOLMES experiment is very challenging, due to the fact, that unlike $^{187}$Re, $^{163}$Ho Q-value and its production need to be determined. Therefore, the first two steps needed to demonstrate the feasibility of a Ho experiment are the accurate determination of the Q-value of $^{163}$Ho EC decay and the production of $^{163}$Ho. The last two steps in HOLMES will be, the detector performance and the theoretical uncertainties.

$^{163}$Ho Q-value

The sensitivity of the $^{163}$Ho neutrino mass experiment strongly depends on the Q-value of the decay, since the smaller Q-value means M peaks are in closer proximity to the end-point energy, and better the statistical sensitivity. As the Q-value increases, the gap between the M peaks and the end-point energy enlarges, resulting in deteriorated experimental sensitivity.

Previously determined Q-values relay on the capture ratios, which are affected by large uncertainties due to errors in the theoretical atomic physics factor and the correlation with the neutrino mass [15, 100]. Therefore, the experimentally determined Q-values found in literature are within the interval of 2.2 to 2.8 keV. This energy range of the Q-value affects the achievable neutrino mass sensitivity by more than a factor of three. An accurate measurement of Q-value is necessary for a better estimation of the sensitivity of HOLMES.

Additional effects that potentially alter the $^{163}$Ho EC spectrum must be investigated and implemented to obtain an unambiguously complete expected spectrum. As suggested by Rujula and Lusignoli [58], a high energy resolution and high statistics calorimetric measurement of the total absorption spectrum is an urgent necessity for neutrino mass measurement that uses the end-point of Ho EC decays.

A calorimetric end-point measurement is the most straightforward approach to determine an accurate Q-value of Ho EC decay. Any possible Q-value between 2.2 to 2.8 keV can be measured with accuracy better than 20 eV by using a single microcalorimeter with about $5 \times 10^5$ counts. The required energy resolution of the microcalorimeter used in Q-value measurement is about 10 eV, which is an easily achievable limit. If the count rate remains less than 100 counts per second, then the measured Q-value is almost unchanged by the detector pile-up, and thus the total statistics can be converted directly to total time. A most recent Q value obtained from a prototype microcalorimeter is $2.80 \pm 0.08$ KeV [99].
$^{163}$Ho Production

$^{163}$Ho is not available directly from nature and large scale production of high purity Ho isotope is required for neutrino mass experiments. A reliable production of $^{163}$Ho with negligible radioactive impurities requires dedicated resources. There are at least three different methods that are the most appealing for the production of the $^{163}$Ho isotope as required by the neutrino mass experiments [7]:

- Neutron activation in nuclear reactor of $^{162}$Er;
- Alpha –particle bombardment of $^{165}$Ho target;
- Gamma –particle bombardment of $^{165}$Ho target.

Identifying the best method of production of $^{163}$Ho is one of the milestones for HOLMES. The production of $^{163}$Ho using the neutron activation of 38% $^{162}$Er enriched sample of erbium oxide has been confirmed [101]. The irradiated sample was preserved for one month to get rid of short-lived radioactivity. After verifying the absence of both short and long-lived radioactive contaminations, the sample was dissolved in a hydrochloric acid solution and then deposited onto the absorber of a TES microcalorimeter. Analysis of the observed spectrum verified the presence of $^{163}$Ho, as shown in Fig. 4.5. The measured calorimetric spectrum, shows structures of the EC decay of $^{163}$Ho with few atypical features [102]. The presence of $^{163}$Ho in the solution was also confirmed at the University of Milano-Bicocca using Inductively Coupled Plasma Mass Spectrometry [7]. A large scale production, purification, and testing are being done at the 1 MW Portuguese Research Reactor (RPI). The reactor is at the nuclear and Technological Institute in Lisbon and it has estimated production capacity of about 3 kBq/mg ($^{162}$Er) per week [101].

![Fig. 4.5: Observed Calorimetric EC decay of $^{163}$Ho spectrum using a thin layer $^{163}$Ho compound onto Bismuth-Iridium microcalorimeter [101].](image-url)
Detector Performance

Energy resolution, events rise time, decay time, and read-out electronics must be considered carefully in designing a microcalorimeter for the application of neutrino mass experiments, as discuss in the previous chapter.

In HOLMES, the construction and optimization of TES detector with Ho embedded absorber is under investigation. For source embedded absorbers the requirements are:

- Potential to embed holmium isotope;
- Very high conductivity or diffusivity at working temperature;
- Good stopping power to absorb the decay energy in the limited volume of the absorber;
- Sufficiently low heat capacity at the working temperature after the $^{163}$Ho source is embedded;
- Efficiency and reproducibility in fabrication, as $10^3$-$10^4$ detectors are necessary.

To maximize the energy resolution for large pixel TES microcalorimeters a simulation of the performance of a possible microcalorimeter design was performed [103]. For minimal degradation of the resolution of the devices with a typical design, in which a small TES is placed at the center of the pixel with a large in-plane absorber around the TES, the diffusivity of the absorber must be higher than $1\times10^5 \mu m^2/\mu s$ (residual resistance ratio (RRR) > 6) [103]. This simulation analysis also showed that energy resolution of 2 eV can be achieved with high-quality gold films for the absorber (RRR>6). At working temperature of a TES detector, 0.1K, gold film possesses volumetric capacity $C = 7.14 J K^{-1} m^{-3}$ and thermal conductivity $G = 5.72 W K^{-1} m^{-1}$ [44]. High conductivity and corresponding short rise time of a gold absorber are the requirements for a Holmium experiment, to minimize the pulse pile-up effect.

The single pixel prototype, in HOLMES, must meet the following design requirements [13]:

1. an energy resolution of 1 eV FWHM at 2 KeV,
2. a rise time of about 1 μs,
3. an ETF reduced fall time of few tens of μs,
4. a 100 μm × 100 μm metallic absorber (Au, Cu or Bi) to be grown in two steps, before and after the $^{163}$Ho implantation, in order to fully embed the source for complete absorption of the emitted radiation,
5. provide all the wiring for the detector bias and for the inductive coupling to the readout dc-SQUID.

Different techniques for embedding the source in the detector are currently being explored. The next chapter will focus on the implantation parameters required to achieve a high concentration of Ho and Er in a gold absorber to then, explore the thermal properties of the Au absorber, and see how this may affect the detector resolution.

Statistical Sensitivity a $^{163}$Ho Experiment

The statistical sensitivity of a holmium experiment has been worked out in details by Galeazzi et al. [7]. A frequentist Monte Carlo code has been developed to approximate the statistical sensitivity of neutrino mass experiments carried out with microcalorimeters [101]. In the Monte Carlo analysis, many Ho EC decay energy spectra, characterized by the same experimental set up, are simulated and then fitted with real ones. The distribution of the acquired $m_\beta^2$ parameters is then used to obtain statistical sensitivity. The total statistics $N_{ev}$, the detector energy resolution $\Delta E_{FWHM}$, the fraction of unresolved pile-up events $f_{pp}$, and the background $b$ are considered as the Monte Carlo parameters characterizing the experimental set up. These input parameters can be expressed in terms of the actual parameters describing a real experiment such as:

$$N_{ev} = N_{det} A_\beta t_m = TA_\beta$$

$$f_{pp} \approx A_\beta \tau_R$$

where $N_{det}$ is the number of detectors, $A_\beta$ is the Ho EC decay activity of a single detector, $t_m$ is the running time of experiment, $T = N_{det} t_m$ is the exposure, and $\tau_R$ is the pile-up resolving time. The simulated energy spectrum $S(E)$, which is expected to be measured by virtual experiments, can be
expressed as [7]:

\[ S(E) = \frac{N_{ev}}{\lambda_{EC}} \int_0^{2(Q - m)} dE_1 R(E - E_1) \times \left( \frac{d\lambda_{EC}}{dE_1} (E_1) + f_{pp} \int_0^{E_1} dE_2 \frac{d\lambda_{EC}}{dE_2} (E_2) \right) \] (4.5)

where \( d\lambda_{EC}/dE_1 \) is expressed in Eq. 2.53, the background \( b(E) \) is assumed to be zero and the response function \( R(E) \) is assumed to be a Gaussian function of the following form:

\[ R(E) = \frac{1}{\sigma \sqrt{2\pi}} e^{(E^2/2\sigma^2)} \] (4.6)

The mean standard deviation is given by \( \sigma = \Delta E_{FWHM}/2.35 \). The simulated spectra are fitted using Eq. 4.4 and free parameters such as \( N_{ev}, f_{pp}, Q \)-value, and \( m_\nu^2 \). Various results from Monte Carlo simulations, were carried out for different \( Q \)-values (2200 eV, 2400 eV, 2600 eV, and 2800 eV). The dependence of the neutrino mass sensitivity as a function of total statistics \( N_{ev} \) was simulated and showed that there is significant improvement of the sensitivity with the increase in statistics. For example, for an energy resolution of \( \Delta E_{FWHM} = 1 \) eV, a pile-up fraction of \( f_{pp} = 10^{-5} \), and a \( Q \)-value of 2.6 keV, the total statistics required to achieve a sub-eV sensitivity on neutrino mass, i.e. \( m_\nu \approx 0.2 \) eV, is the order of \( 10^{14} \).

Also, the dependence of the neutrino mass statistical sensitivity on the energy resolution \( \Delta E_{FWHM} \) and on the pile-up events \( f_{pp} \) fraction was simulated, and it showed that the sensitivity degrades rapidly with an increase of \( f_{pp} \), while the detector energy resolution has weak impact on the sensitivity of a Ho EC decay neutrino experiment.

The statistical sensitivity was plotted against the single detector activity \( A_B \) for four different \( Q \)-values and two different sets of detector parameters, as shown in Fig. 4.6. The lower panel corresponds to a configuration with \( \Delta E_{FWHM} = 1 \) eV , \( \tau_R = 1 \mu s \), \( T = 10^5 \) detector \times year , for the detector parameters. In this configuration, the experimental exposure of \( 10^5 \) could be achieved by operating \( 10^4 \) detector arrays for 10 years. The upper panel represents a more challenging configuration, in which the energy and time resolution are improved by factor of 10 to obtain experimental exposures that are a factor of 10 higher. From Fig. 4.6 one can conclude that, for a given energy resolution \( \Delta E \), pile-up resolving time \( \tau_R \), and experimental exposure \( T \), the statistical sensitivity significantly improves with the increase in single detector activity. Furthermore, the increase in detector activity not only increases the experimental statistics but also the unresolved fraction of pile-up events. The resulting effect, however, is an enhancement in the statistical sensitivity of the experiment. About \( 2 \times 10^{11} \) nuclei of \( ^{163}\text{Ho} \) isotope can produce activity of about one decay/sec. The statistical sensitivity improves with the decrease in the \( Q \)-value but the increase in sensitivity is not consistent with the decrease in \( Q \)-value.

Table 4.3 shows the calculated experimental exposure necessary to achieve neutrino mass statistical sensitivities of 0.2, 0.1, and 0.05 eV for the highest and lowest possible \( Q \)-values.
Configuration I corresponds to the detector parameters $\Delta E_{\text{FWHM}} = 1 \, \text{eV}$, $\tau_R = 1 \mu s$, $A_\beta = 1000 \, \text{Hz}$, while configuration II refers to parameters $\Delta E_{\text{FWHM}} = 0.3 \, \text{eV}$, $\tau_R = 0.1 \mu s$, $A_\beta = 10000 \, \text{Hz}$.

<table>
<thead>
<tr>
<th>Q (keV)</th>
<th>Target Sensitivity (eV)</th>
<th>Exposure time [detector×years]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Config. I</td>
</tr>
<tr>
<td>2.2</td>
<td>0.2</td>
<td>$2.6 \times 10^4$</td>
</tr>
<tr>
<td>2.2</td>
<td>0.1</td>
<td>$4.1 \times 10^5$</td>
</tr>
<tr>
<td>2.2</td>
<td>0.05</td>
<td>$6.6 \times 10^6$</td>
</tr>
<tr>
<td>2.8</td>
<td>0.2</td>
<td>$6.5 \times 10^6$</td>
</tr>
<tr>
<td>2.8</td>
<td>0.1</td>
<td>$1.0 \times 10^8$</td>
</tr>
<tr>
<td>2.8</td>
<td>0.05</td>
<td>$1.7 \times 10^9$</td>
</tr>
</tbody>
</table>

Table 4.3: The targeted sensitivities and required experimental exposure for two extreme Q-values and different sets of detector parameters [7].
5 Holmium Implantation and Analysis of Gold thin films

As already discussed in Chapter 4, it is really important in a Holmium Experiment to optimize the detector parameters such as, energy resolution, rise time and decay time. The variable that all these parameters have in common is the heat capacity, C.

Heat capacity depends on the absorber material of the TES, and an important question that needs to be answer is if when Holmium and Erbium are implanted in the absorber, does the heat capacity of the absorber changes?

In this Chapter will be explore the properties of gold thin films, since as mentioned in Chapter 4, due to its high conductivity is a promise candidate for HOLMES experiment, when embedded with holmium and erbium. Although the goal is to implant Holmium and Erbium radioactive, the radioactivity does not change thermal properties such as heat capacity, and so to simplify, this experiment was done with Ho and Er non-radioactive. Furthermore, due to the fact of the process of $^{163}$Ho fabrication using $^{162}$Erbium is still being studied, and it requires a lot of time for the purification processes, in this experiment the isotopes used were $^{163}$Ho and $^{166}$Er. Again, to simplify the experiment and since the atomic mass unit does not interfere with the parameters which need to be optimized, less expensive isotopes are easily commercialized were used.

These results will then be useful to conclude if Ho$^+$ and Er$^+$ implanted gold films can be used as potential candidates for source-embedded absorbers for a Holmium experiment.

5.1 Gold Thin Films Fabrication

In this experiment the samples consisted of gold, as absorber, and a silicon substrate. For the adhesion of the absorber on the substrate an iridium layer was used.

The gold was deposited via thermal evaporation, on a 60 nm thin iridium, Ir, sticking layer produced with Pulsed Laser Deposition, PLD, on the top of a silicon wafer. These samples were prepared at the University of Genoa, Italy, and, consisted of 2 μm thick gold, Au, film deposited on 250 μm thick (111) silicon, Si, wafer 1 cm×1 cm.

The deposition apparatus in Genoa University is formed by three vacuum chambers, communicating between them: one where the samples are inserted, another for the Pulse Laser Deposition (PLD) and the last chamber dedicated to the electron gun. Each of these rooms has an independent pumping, composed by a rotative pump, low vacuum, ~$10^{-2}$ mbar and a turbo-molecular pump for high vacuum, ~$10^{-8}$ mbar. The advantage of this system is to allow the fabrication of a multilayer film using different techniques, under conditions of ultra-high vacuum, leaving the interfaces free from contamination.

To ensure a good homogeneity and a proper growth of the film the PLD technique is used, for the thin iridium layer. PLD technique is a thin film deposition technique where the pulsed laser beam of high power and of 1064 nm, is focused inside the vacuum chamber to achieve a material surface area of 0.1 mm². The material being deposited, in this case, is the a monocrystal of iridium. This material is vaporized from the target, in a plasma column which deposits as a thin film on the substrate. When a laser pulse is absorbed by the target, energy is first converted to an electronic excitation and then to thermal, chemical and mechanical energy, resulting in evaporation, ablation, and plasma formation. An important characteristic of this technique is that only a very small portion of the target of iridium is heated, reducing the risk of film contamination from the oven. Moreover the plasma of Ir generated by the laser is so energetic that allows a very good adhesion between the substrate and the film, so as to create at the interface between the wafer and an the adhesiom film, Ir - Si.

For the gold layer the thermal evaporation is used. This technique is a process where the solid material is heated inside a chamber in high vacuum, ~$10^{-5}$ Pa, at a temperature that generates a vapor pressure. After heating the material, a vapor stream is formed, reaching the object to be coated, the substrate. In this technique the heat source is a filament. The advantages of the thermal evaporation are a
high deposition rate (~0.5 \( \mu \text{m/min} \)), no need to heat the substrate, and the low cost. The non-uniform coverage of the substrate, the poor step coverage, and the contamination of the deposited film due to the evaporation of the chamber material, are the main limitation of this technique.

5.2 Ho and Er Implantation and Characterization

5.2.1 Implantation Process

The implantation is done through ionized beams of particles that are accelerated with energies between keV and MeV. This process induces changes in the solid properties in two ways: by compositional changes due to the introduction of impurities (doping) or by structural changes caused by the energy transfer of ions (projectiles) to the target.

During the interaction of the beam with the target, various phenomena that stop or scatter the incident particles can occur. The main ones are:

- Inelastic collisions with the electrons of the binding medium, wherein the energy lost in the collision is directed to the ionization or excitation of atoms or molecules;
- Inelastic collisions with the nuclei, which can promote nuclear excitations and nuclear reactions;
- Elastic collisions with the electrons of the binding medium;
- Elastic collisions with the nuclei, where the energy is transferred to the particles that absorb the impact.

Nuclear inelastic collisions and electron elastic collisions play an insignificant role in stopping particles, compared with the electron inelastic collisions (electronic stopping power) and nuclear elastic collisions (nuclear stopping power). The prevalence of a given stop mechanism, nuclear or electronic, is a function of the mass and energy of the accelerated particles and of the atomic mass of the medium.

In an implantation process it is important the energy lost of the beam particles as they pass through the material under study, since this energy lost depends on the type and energy of the radiation and on the properties of the material. The nuclear stopping power, \( S_n \), and electronic stopping power, \( S_e \), can be defined by the following expression [104]:

\[
S_{n,e} = -\frac{1}{N} \left( \frac{dE}{dx} \right)_{n,e}
\]  
(5.1)

where \( N \) is the atomic density of the medium, and \( dE/dx \) is the the energy loss as a function of the distance.

The total stopping power is given by:

\[
S = S_n + S_e
\]  
(5.2)

The variation of both stopping powers as a function of the reduced energy, as shown in Fig. 5.1, indicates that for low energies, typically on the order of keV, the nuclear stopping effect predominates, while for higher energies values (~MeV) the nuclear stopping power becomes negligible compared to electronic stopping power.
Typically, the energy interval used for ion implantation processes requires accounting for both stopping effects [104]. Assuming that both processes are independent, one can define the energy loss per unit distance by [105]:

\[- \frac{dE}{dx} = N|S_n(E) + S_e(E)| \quad (5.3)\]

For known \(S_n(E)\) and \(S_e(E)\) values, it is possible to integrate Eq. (5.3) to obtain the path of the implanted ions [105]:

\[ R = \int_0^E \frac{dE}{S_n(E) + S_e(E)} \quad (5.4) \]

where \(R\) is the average distance traveled by a particle of energy \(E\), in an amorphous medium.

The concentration profile versus distance, result of an ion implantation process, approximately follows a Gaussian distribution [105]:

\[ N(x) = \frac{N_i}{\sqrt{2\pi}\Delta R_p} \exp \left( - \frac{(x-R_p)^2}{2\Delta R_p^2} \right) \quad (5.5) \]

where \(R_p\) is the average distance of the ions, \(\Delta R_p\) is the standard deviation, and \(N_i\) is the implanted dose.

The maximum doping concentration is given by [105]:

\[ N_{MAX} = \frac{N_i}{\sqrt{2\pi}\Delta R_p} \quad (5.6) \]

**Implantation Experimental Setup**

The experiments reported in this work were carried out in *Campus Tecnológico e Nuclear - IST* facilities. The implantation process was executed with the 210 kV ion implanter, 50 kV in the extraction plus 160 kV on the accelerator tube, schematized in Fig. 5.2. This implanter allows the implantation of an area of 20x20 cm² over a range of temperatures from 77 to 1273 K, and currents on target up to 10 mA [106]. The extracted beam at up to 50 keV is mass analyzed in a double focussing 90° analyzing magnet with mass resolution of \(M/\Delta M\approx150\) to 250 [106]. The implanter is coupled to a vacuum system with a diffusion and a turbomolecular pump, guaranteeing a high vacuum, monitored with a Pirani gauge (\(p > 10^{-3}\) mbar) and a Penning gauge (\(p \in [10^{-3}, 10^{-7}]\)
mbar). The typical pressure in the implantation chamber is in the $10^{-7}$ mbar range and the working pressure during implantation is $2 \times 10^{-6}$ mbar.

In this work, 4 Au(Si-Ir) samples were used: one reference (Au) and three other implanted, where one was implanted with holmium (AuHo), another with erbium (AuEr) and the last one with holmium and erbium (AuHoEr).

The first implanted sample was the Ho one using a voltage extraction of 30 kV and a voltage acceleration of 95 kV. The beam energy used was 250 keV with a beam current of 25 $\mu$A. In this sample the goal was to implanted a Ho fluence of $8E+16$ at/cm$^2$. This first implantation process had a total duration of 2h33min48s. The maximum energy of the implanter is 210 keV, to achieve an energy of 250 keV a potential of 125 kV was used with Ho$^{+4}$, as $E = qV$, where $E$ is the energy, $q$ is the charge and $V$ the potential.

During implantation, a change in color of the sample was observed through the implantation camera window. This modification was not expected, accounting for 12 previous experiments [44]. The sputtering coefficient of Ho and Er in Au was simulated using the SRIM program [108]. For SRIM simulation the input parameters where: implanted ion, its energy and angle of incidence, other elements in the sample, and their density. The simulation enables to obtain the medium-range of the ions. The second Ho implantation process was done in 3h50min41s with a Ho fluence of $2.4E+16$ atm/cm$^2$. The implantation with Er had a total duration of 5h04min23s and was performed simultaneously in two samples with the arrangement shown in Fig. 5.3, since the Er fluence of $5.7E+16$ atm/cm$^2$ was the same for both samples. This process was executed in two stages, since the first implantation parameters were not reachable for shorter periods. Second day implantation process continued with an Er fluence of $2.3E+16$ atm/cm$^2$.
Simulation of Ions Profile

SRIM program showed that the medium-range of Ho ions with an energy of 250 keV in Au thin film is of the order of 25 nm, as in Fig. 5.4.

The simulation with the SRIM program, considering an energy of 250 keV with an atomic mass of 165 amu for Ho, resulted in a sputtering coefficient, S, of 30.86 atoms/ion in Au thin film. This means that for each ion of Ho implanted about 31 atoms of Au are removed from the sample.

For a sputtering coefficient of ~30 and a fluence of $8 \times 10^{16}$ at/cm², the sputtering of Au is $2.4 \times 10^{18}$ at/cm² or approximately 400 nm.

A Au film having a thickness of $L=3000$ nm, after sputter removal by implantation with a fluence of $8 \times 10^{16}$ at/cm² and an energy of 250 keV for the Ho ions:

- The film should become $L' = 2600$ nm thick;
- A retained Ho fluence of $4.8 \times 10^{15}$ at/cm², approximately 6% of its nominal fluence.

If the implantation fluence was 10 times as low, as in the 12 previous experiments [44], $8 \times 10^{15}$ at/cm², the corresponding quantities would be $L' = 2960$ nm and a retained Ho fluence of $4.7 \times 10^{15}$ at/cm², approximately 58% of its nominal fluence.

For an energy of 180 keV the Ho sputtering in Au was also simulated, and Er in Au, using the same conditions as for Ho, the sputtering coefficient was obtained. The results of these simulations are shown in Table 5.1.

<table>
<thead>
<tr>
<th>Sputtering Coefficient</th>
<th>Element</th>
<th>E = 180 keV</th>
<th>E = 250 keV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Element</td>
<td>31.18</td>
<td>30.86</td>
<td></td>
</tr>
<tr>
<td>Ho</td>
<td>31.12</td>
<td>31.06</td>
<td></td>
</tr>
</tbody>
</table>

Table 5.1: Sputtering coefficient SRIM simulation results for Ho and Er in Au thin film with energies of 180 and 250 keV.
The simulation results indicated that the implantation fluence needed to be changed, using the extraction and acceleration voltage as the first implanted sample, and also the same beam energy.

5.2.2 Thin Film Characterization Techniques

Rutherford Backscattering Spectroscopy (RBS)

The Rutherford Backscattering Spectroscopy, RBS, technique, schematized in Fig. 5.5, is an ion scattering technique used for compositional thin film analysis by measuring the backscattering of a high energy ion beam impinging on a sample [109]. The target is ion bombarded with an energy in the MeV-range (typically 0.5-4 MeV), and the energy of the backscattering projectiles is recorded with an energy sensitive detector, typically a solid state detector. RBS allows a quantitative determination of the composition of the sample material and depth profile of individual elements. Since the backscattering cross section for each element is known, it is possible to obtain a quantitative compositional depth profile from the RBS spectra. The partial energy transfer from an incident particle to a target particle is given by the kinematic factor, $K$, which relates the energy of the incident particle $E_0$ with the energy $E_1$ after the collision by $E_1 = KE_0$ [111]:

$$K = \frac{M_1^2 - M_1^2 \sin^2 \theta^2 + M_4 \cos \theta}{M_1 + M_2}$$

(5.7)

where $M_4$ is the mass of the projectile, $M_2$ is the mass of the target, and $\theta$ is the backscattering angle. The energy and angle of the backscattered particle are obtained experimentally, and the value of $M_2$, being the only unknown parameter, is then calculated. The measurement and study of the energy lost as a function of distance allows the determination of the concentration versus depth. Considering the value of $dE/dx$ constant, valid assumption for the RBS analysis since it is a surface technique and, in general, the beam energies are near or above the maximum of the stopping power curve, and considering the trajectory of the ion throughout the sample, one obtains [112]:

$$\Delta E = \left[ \frac{K}{\cos \theta_1} \frac{dE}{dx_{E_0}} + \frac{1}{\cos \theta_2} \frac{dE}{dx_{KE_0}} \right] x$$

(5.8)

where $\theta_1$ is the angle between the incoming beam and the normal to the sample surface, $\theta_2$ the angle between the outgoing beam and the normal to the sample surface, $KE_0$ is the energy right after surface collision and $x$ the depth at which the backscattering occurs. Terms $dE/dx_{E_0}$ and $dE/dx_{KE_0}$ will be the stopping power at an energy $E_0$ and the stopping power at an energy $KE_0$, respectively. Note the negative sign in the stopping power definition, generates a positive value for the stopping power, since the energy decreases as $x$ increases. Simplifying the expressions, one can write [111]:

$$\Delta E = [S] x$$

(5.9)

The stopping power in multi-element regions is assumed to be that obtained by weighting the individual stopping powers with the element’s concentration (Bragg’s rule). From Eq. 5.9 it is possible to determine the depth at which the backscattering occurs. For the beam energy used in RBS, the scattering is Rutherford (pure Coulomb scattering) then the general form of Rutherford cross section is used to compute the probability of a backscattering event [111]:

46
where \( \frac{d\sigma}{d\Omega} \) is the scattering cross section per unit solid detection angle, \( z_1 \) is the atomic number of projectile, \( z_2 \) is the atomic number of target, \( M_1 \) is the mass of projectile, \( M_2 \) is the mass of target, \( e \) is the electronic charge, \( E \) is the projectile energy, \( \theta \) is the backscattering angle, and the subscripts 1 and 2 refer to projectile and target, respectively. The Rutherford cross section is proportional to \( Z_2^2 \), as shown in Eq. 5.10, which means that RBS has a higher sensitivity to heavier elements. The number of particles detected by the detector positioned at scattering angle \( \theta \), \( Q_D(\theta) \), is given by [112], and schematized in Fig. 5.6:

\[
Q_D(\theta) = \Omega \sigma(\theta) Q N t
\]

where \( N \) is the atomic density of the sample, \( t \) is the thickness of the sample, \( Q \) is the total number of incident particles, \( \sigma(\theta) \) the scattering cross section given by [111]:

\[
\sigma(\theta) = \frac{1}{\Omega} \int \frac{d\sigma}{d\Omega} d\Omega
\]

and \( \Omega \) is the solid angle \( \Omega = \frac{a^2}{l^2} \), where \( a \) is the effective detector area, and \( l \) is the distance between the detector and the sample. In Eq. 5.11, \( N t \) is the atomic density in the sample in \( \text{atoms/cm}^2 \). To infer \( N t \) from \( Q_D(\theta) \) one needs to know the scattering cross section \( \sigma(\theta) \). The scattering cross section can be calculated from the force that acts during the collision, since high energy particles penetrate up to the core of the target atom, given by Eq. 5.10. This force mainly corresponds to an unscreened Coulomb repulsion between the two positively charged nuclei. In a RBS spectrum, the height, \( H \), of the count number in a multi-spectrum channel, \( k \), is the number of events with energy between \( E_k \) and \( E_k - \delta \). Eq. 5.9 associates to each channel \( k \), with a width \( \delta \), a layer of the sample with a thickness \( x = \delta /[S] \). Thus, the number of events collected in a channel \( k \), \( H_k \), is a function of the associated sample layer thickness and of the number of spreader units (atoms of specie “i”) in the layer. From Eq. 5.6, \( H_k \) is given by [113]:

\[
H_k = \sigma_i(E_k).\Omega. Q. N_i. \left( \frac{x_i}{\cos \theta_1} \right)
\]

where the thickness was corrected by the factor \((\cos \theta)^{-1}\) since the incident angle is not normal to the surface. Replacing \([S_0]/N = [\varepsilon_0]\) and \( x = \delta /[S] \) in Eq. 5.8, where \([\varepsilon_0]\) is defined as \( \varepsilon = (1/N) [S] \), and assuming valid the surface approach [113]:

\[
H = \sigma_i(E_0).\Omega. Q. n_i. \left( \frac{\delta}{[\varepsilon_0]\text{medium}[\cos \theta]} \right)
\]

where \([\varepsilon_0]\) is the stopping power calculated as in Eq. 5.8, \( n_i = N_i/N \) is the atomic fraction of element \( i \) in the sample with a total atomic density \( N = \sum N_i \sigma(E_0) \) is the average differential cross section (averaged over the solid angle \( \Omega \)) calculated for the \( i \) element and a beam with energy \( E_0 \), and \( \delta \) is the energy range defined by a channel (keV/channel) which is determined by the detection electronics. In the case where there is more than one element present on the sample surface, each will produce a signal with a surface height \( H_{0i} \), in their respective energy \( E = K_i E_0 \). For two elements, A and B, with atomic densities, \( n_A \) and \( n_B \) respectively [113]:
\[ \frac{H_{0A}}{H_{0B}} = \frac{\sigma_A(E_0) n_A [e_{0}]_{\text{medium}}}{\sigma_B(E_0) n_B [e_{0}]_{A}} \]  

Eq. 5.15 gives allows to obtain \( n_A / n_B \), the ratio of atomic concentrations of the elements A and B in the sample. In zero-order approximation, one can assume \( [e_{B}] / [e_{A}] = 1 \) and calculate \( (n_A / n_B)_\circ \). With this value is possible to estimate \( [e_{B}] / [e_{A}] \) and refine the value of \( n_A / n_B \). A single iteration produces generally satisfactory results. The ratio \( H_A / H_B \) is independent of the width \( x_i \), different for each element, and of the atomic density of the sample, depending only on the ratio of the atomic fractions constituting the film.

**Particle-Induced X-ray Emission (PIXE)**

Particle Induced X-ray Emission, PIXE, resumed in Fig. 5.7, is an analytical method which relies on the spectrometry of characteristic X-rays emitted by the target elements due to the irradiation of a high energy ion beam (typically 1-2 MeV, of H or He), used to ionize inner electron shells (K or L) of the atoms in the sample [114]. Since there is little overlapping of the characteristic X-rays for different elements, simultaneous detection of complicated multi-elements sample is possible. Bombardment with ions of sufficient energy produced by an ion accelerator, will cause inner shell ionization of atoms in a specimen. Outer shell electrons drop down to replace inner shell vacancies, however only certain transitions are allowed. X-rays of a characteristic energy of the element are emitted and measured by an energy dispersive detector, where the intensities are then converted to elemental concentrations.

The continuous of X-rays in Fig. 5.8, is the radiation emitted by the electrons that interact with the sample, being decelerated by the present charges, resulting in a background radiation which affects the spectral resolution of the spectra. The accelerating ions occasionally interact with the electrons of the innermost layer of the atoms in the sample, resulting in ejected orbital electrons, as shown in Fig. 5.8. The remaining electrons are redistributed, causing the outer layers electrons take the place of the ejected orbital electrons. In this case, the outer layer electrons lose energy in order to occupy a lower energy state, which can be released in the form of a photon of characteristic X-rays, or that lost energy can be transfer to the valence electrons causing one of them to be ejected, called Auger electrons. PIXE methodology enables an increase in the detection sensibility of chemical elements by analyzing their characteristic X-rays, mainly due to the fact of the incident particles having a higher mass than the electron. In this way, a proton beam loses less kinetic energy, having a higher capacity to penetrate the sample than an electron beam. Furthermore, due to their higher mass, particles are much less decelerated than the electrons resulting in a much reduced Bremsstrahlung and thus become much easier detection of X-ray characteristic lines of the elements present in the sample.

For characteristic X-ray production is necessary to remove electrons from the atoms inner layers, for example the K-layer. For that, the minimum energy must exceed the binding energy of electrons in that layer, called the electron binding energy or cutting absorption energy.
For X-rays detection, the detectors typically used are Si(Li) detectors, since its detection efficiency and energy resolution are very suitable for the desired energy range (5-20 keV). Limited by the absorption of the detector window, PIXE method is capable of identifying and quantifying elements with Z > 10, with a detection limit of \(10^{-6} \text{g/g (ppm)}\), or \(10^{14} \text{at/cm}^2\) in case of thin films, and an absolute precision of 5 to 30%. Samples can be small, up to a mass limit of approximately 1 ng.cm\(^{-2}\)[113]. The quantitative analysis, in thin or thick samples, is usually carried out from programs that access cross sections libraries or other parameters to determine the concentrations of various elements in the sample. In PIXE analysis of a thin film, the number of detected X-rays, \(N_x\), is proportional to the amount, \(t_z\), of the chemical species with atomic number, \(Z\), present in the sample (at/cm\(^2\)) and to the collected charge \(Q\) [113]:

\[
N_x(Z) = \frac{\Omega}{4\pi} \cdot \sigma_x \cdot \xi \cdot T \cdot \frac{q}{q_e}
\]  

(5.16)

where \(\Omega\) is the solid angle subtended by the detector, \(\sigma_x\) is the cross section of characteristic X-rays production, \(\xi\) is the detector efficiency, \(T\) is the transmittance of the X-rays of the sample for the absorbers placed between the sample and the detector, \(q\) is the charge of the incident particle, \(e\) is the electron charge, and \(t_z\) is the number of atoms of specie \(Z\) per unit area. Eq. 5.16 can be rewritten defining the effective yield for X-rays by \((\text{at/cm}^2)\) per unit of incident charge by [113]:

\[
Y_{ef}(Z) = \frac{\Omega}{4\pi} \cdot \sigma_x \cdot \xi \cdot T \cdot \frac{1}{q_e}
\]  

(5.17)

The number of atoms of specie \(Z\) per unit area as [113]:

\[
t_z = \frac{1}{Y_{ef}} \left( \frac{N_x}{Q} \right)
\]  

(5.18)

From Eq. 5.18 is possible to determine the elemental concentration in the sample, given the number of detected X-rays and the incident charge used. As \(Y_{ef}\) depends only on the parameters of the experimental instrumentation and fundamental physical quantities, in practice, \(Y_{ef}\) is determined with a calibration using samples of known thickness and Eq. 5.18, where \(t_z\) is a known parameter.

The X-ray spectrum is essentially defined by peaks or stripes related to the X-rays characteristic of the elements present in the sample. Besides the contribution of the X-rays of the elements present in the sample, there are other contributions which are also present in the spectrum, in particular, the sum peaks, escape peaks and background radiation. The sum peaks result from the simultaneous detection of two or more X-ray photons, appearing in the spectrum at a position corresponding to an energy equal to the sum of the energies of the photons detected in coincidence. The escape peaks arise due to ionization of Si K-layer in the detector, in which results the emission of 1.740 keV photons that sometimes escape from the crystal, losing therefore the corresponding energy. Under these conditions, the energy deposition is not complete, and in the X-rays spectrum also appears a escape peak, with an energy difference of 1.740 keV from the characteristic photoelectric peak. The contribution of this effect is less than 5%. The background radiation results mainly due to three effects: the ejected electrons deceleration (secondary electrons), the projectile deceleration and the occurrence of nuclear reactions. Any of these contributions gives rise to a continuous spectrum that overlaps with the characteristic X-ray spectrum. A large percentage of nuclear reactions that contribute to background of PIXE spectra, do not occur only at the target, but also in the collimators of the beam transport lines or in other components of the sample chamber.
X-ray Diffraction (XRD)

X-ray diffraction, illustrated in Fig. 5.9, is a technique typically used for the study of crystal structures and atomic spacing. X-rays have a wavelength that is similar in magnitude to the spacing between the atoms in most inorganic materials, between 0.1-10 Å [116]. The wavelength of an X-ray radiation is thus of the same order of magnitude as the lattice constants of crystals, and it is this which makes X-rays useful in the analysis of crystal structures. In materials with regular structure (i.e. crystalline), the scattered X-rays undergo constructive and destructive interference, being diffracted by crystal lattices. The directions of possible diffractions depend on the size and shape of the unit cell of the material, thus an examination of the X-ray diffraction patterns, meaning the intensities of the diffracted waves, produced yield information regarding the arrangement of atoms within the lattice. Any material will produce a diffraction pattern characteristic of its compounds or phases, leading to a phase identification.

The diffraction of X-rays by crystals is described by Bragg’s Law, shown in Fig. 5.10, which relates the wavelength of electromagnetic radiation (λ) to the diffraction angle (θ) and the lattice spacing (d) in a crystalline sample. The path difference between the two rays is just 2dsinθ, where θ is the angle of incidence, which is conventionally measured from the plane of “reflection”. From Fig. 5.10, one can notice that θ is just half the angle of detection of the incident beam.

For the rays to interfere constructively, the path difference must be an integral number of wavelengths, leading to the Bragg condition:

\[ 2d \sin \theta = n\lambda \]  \hspace{1cm} (5.19)

where n is an integer, known as the order of the corresponding Bragg reflection. The planes in a crystal are characterized by the miller indexes h, k, and l. A Miller index for a plane is expressed as (hkl), where h, k and l are integers such that the shortest distance between Bragg planes is inversely proportional to the index. In order to know which Bragg peaks are allowed and to have an idea of their relative intensities it is necessary to introduce the concept of structure factor. This parameter corresponds to the sum of the phases of scattering for all relevant atoms, and so determines the scattering "strength" of a given (hkl) plane. The general expression for the structure factor of a crystallographic plane (hkl) is given by [118]:

\[ F_{hkl} = \sum f_j e^{2\pi i (hu_n + kv_n + lw_n)} \]  \hspace{1cm} (5.20)

where the summation extends over n lattice points \((u_n, v_n, w_n)\), and \(f_j\) is the atomic form factor, which is a measure of the scattering strength of the \(j^{th}\) atom in the unit cell. The d-spacing described by h, k, l indices, characteristic of the unit cell, \(d_{hkl}\), is defined as the pointing vector from the origin of the unit cell to a point perpendicular to the first plane of the family (hkl), defined as [119]:

\[ d_{hkl} = \frac{1}{|d_{hkl}|} \]  \hspace{1cm} (5.21)
For a cubic system, the interplanar distance, considering $a$ the lattice parameter, is given by:

$$d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}}$$  \hspace{1cm} (5.22)

**Analysis Techniques Experimental Setup**

The implanted samples were analyzed using the RBS technique, performed with the 2.5 MV Van de Graaff Accelerator at ITN/IST facilities. The Van de Graaff Accelerator is a type of high-voltage electrostatic generator that serves as a particle accelerator. A high potential difference is built up and maintained on a smooth conducting surface by the continuous transfer of positive static charges from a moving belt to the surface. When used as a particle accelerator, an ion source is located inside the high-voltage terminal. Ions are accelerated from the source to the target by the electric voltage between the high-voltage supply and ground.

The accelerator used is a horizontal electrostatic accelerator, capable of reaching terminal voltages of the order of 3MV. The accelerator is within a metallic cylinder, referred to as tank, which is pressurized with a mixture of two gases in equal proportion, sulfur hexafluoride ($SF_6$) and nitrogen ($N_2$), whose function is electrical insulation, preventing discharge between the terminal and the tank. With this accelerator beams of hydrogen ($H^+$) and helium ($He^+$) ionized may be produced. Normally, the lines and chambers are usually at a lower pressure than atmospheric pressure to prevent dispersion of the ion beam or even the removal of the particles colliding with air molecules. Such pressures are typically less than $2\times10^{-6}$ mbar. Increasing the vacuum is possible to significantly reduce the scattering of the ions by air molecules, thus increasing the mean life of the beam. The pressure measured in the accelerator tube when the beam is accelerated is of the order of $10^{-6}$ mbar. Some of the components that constitute the Van de Graaff accelerator are shown schematically in Fig. 5.11.

![Fig. 5.11: Schematic simplified of the Van de Graaff accelerator [120].](image)

One of the components, the column, has the function of maintaining the electrical and mechanical stability of the terminal, of establishing an electric field to accelerate the ions and of involving the glass accelerator tube, which is kept under vacuum. The high voltage power supply, also called charge source on the belt, is used to inject charges on the surface of the belt. The high voltage source is placed at the reference potential; charges begin to circulate on the belt, and then are collected in the terminal by friction through electrostatic combs. The charge transport allows placing a positive voltage on the terminal, of the order of a few MV, much higher than the one from the high voltage source that is of the order of a few kV. In turn, the movement of the belt will drive
the generator that has the function of feeding the electronic located in the terminal in a very high voltage. The potential difference which is established from the terminal to the ground is subsequently used to accelerate the ions produced in the ion source placed in the terminal. This potential difference is constant, depending on the control parameters of the machine. The terminal voltage depends on the balance between the number of charges that are deposited therein and the number of charges that are removed, according to the charge conservation law. One of the ways to control this voltage is from the leakage current to ground potential due to capacitive coupling between the two potentials. This current is practically negligible, so to control it is used a device called the crown. The crown consists of a movable arm with one end facing the terminal, in which small needles are inserted. When the terminal voltage is constant, the crown current will depend on the distance between the crown and the terminal. It is possible to approximate (crown "In") or move away (crown "Out") the crown through an existing motor outside the tank. In this way, is possible to control the crown current, i.e. the leakage current between the terminal and the ground potential. Varying the distance between the crown and the terminal, it is possible to control the voltage amplitude in the terminal. The crown current is also used to stabilize the terminal voltage.

The ion source is placed inside the terminal, as well as all control components. To produce a plasma one needs to inject gas inside the ion source, which causes an increase of the vacuum reading value, since the gas enters the accelerator tube. To keep the source active and the beam stable, the vacuum reading value needs to be between 10⁻⁶ and 10⁻⁵. The gas ionization inside the ion source is performed based on a radio frequency antenna which operates at 100 MHz with a power of 60 W and is located inside the terminal. The gas is inside two cylinders of He and two cylinders of H. The gas is injected by the action of valves, thermo-mechanical, to create a plasma within the ion source, in which will be confined. Once ionized the gas inside the ion source, the ions are extracted through the application of an electric field that is created between the ends of the source by applying a high voltage, around 2 kV, between the anode terminal and the source output.

After passing through the accelerator tube, the ion beam passes through a electrically insulating circular collimator (so-called iris) with a diameter of about 3 cm. An ammeter connected to the collimator checks the beam straggling. The beam is focused when the iris current is approximately zero. After passing through the circular collimator, the ion beam is controlled in a screen placed in its path (called Tantalus, due to the material it is made). Tantalum is a material with a high melting point, thus is a heat resistant material, and is a good conductor. This target is connected to an ammeter which has the function of measuring the ion beam current. The current reading of the tantalum target allows the user to know if the beam is really focused. When varying the focus, the tantalum current reading varies. The user knows that the beam is focused when the tantalum measured current is maximal.

The beam from the accelerator can be directed to three lines, designated RBS, PIXE and microprobe, as shown in Fig. 5.12a, through a magnetic deflection of a magnet at the accelerator exit, by adjusting the intensity and direction of the field of the deflector magnet. The magnetic field is measured by a NMR magnetic probe. The slits are pieces identical to tantalum targets, placed one on each side of the line, facing each other so that the beam passes through, but slightly touching both slits. Their purpose is to obtain on both slits a current proportional to the lateral deviation of the beam. This current is fed back in the stabilizer, and thus it is possible to maintain a stable beam by controlling the voltage in the terminal [120].

The experimental conditions used for RBS technique consisted of a 1 mm collimated beam of 2.0 MeV 4He⁺ with normal incidence on the samples. The backscattered particles were detected by the three solid state detectors, showed in Fig. 5.12b, where the detectors at 15° and −15° have a resolution of 15 keV and the one at −40° have a resolution of 12 keV. These detectors are displayed in IBM geometry, where the incident beam, exit beam and surface normal of the sample are in the same plane, and they are located inside chamber maintained under vacuum by a turbo molecular pump. Their effective area is 1 cm².
Samples were mounted on a two axes rotation motorized goniometer, allowing steps of 0.04° in φ, in the vertical plane of the sample, and steps of 0.02° in θ, in the horizontal plane of the sample. The samples are within a distance of 15 cm from the detector and they are positively polarized, normally \( V_{pol} \sim 100 \, V \), by means of a series of batteries, in order to minimize the emission of secondary electrons induced by the beam bombardment. The beam current is therefore measured directly from the target. Typically, the beam current is kept below 4 nA, preferably between 2 and 3 nA, in order to minimize the effect of charge accumulation and "pile up" during the measurement. The backscattered ions, after colliding with the detectors generate an electrical pulse proportional to the ion energy. The detector signal is sent to a preamplifier and from this, to a linear amplifier coupled to an analog digital converter (ADC) and to a multichannel analyzer (MCA). The MCA divides the energy region in channels, each representing a small energy interval of the order of keV (typically 1-3 keV). Ions arriving at the detector in each energy interval are summed and the RBS spectrum represents then the number of backscattered particles in each energy interval (channel). The linear correlation between the particle energy and the channel number can be given by [120]:

\[
E = (\#_{ch}\times\varepsilon) + \Delta
\]

where \( \#_{ch} \) is the channel number, \( \varepsilon \) is the channel energy width (keV/channel), and \( \Delta \) is the energy corresponding to the zeroth channel.

The RBS data was analyzed using N. Barradas Data Furnace software (NDF) [121]. NDF is a fortran code, used for the analysis of RBS, ERDA (Elastic Recoil Detection Analysis), PIXE, NRA (Nuclear Reaction Analysis) non-resonant spectra, and data from PIGE (Particle Induced γ-ray Emission), for any ion, target, geometry and spectra number. NDF determines the depth profile that leads to the best fit. This code has an extensive database of scattering cross sections and stopping powers. The inputs of the program are the experimental conditions used and the information on the chemical elements present in the target, in which the amount will be adjusted to give the best fit to the experimental data. The first sample to be analyzed by RBS was the sample implanted with holmium, since it was found through the implantation chamber window a color change of the sample. The remaining three samples, the reference sample, the sample implanted with erbium and holmium and the one implanted with erbium, were analyzed simultaneously.

PIXE was performed using the nuclear microprobe, as shown in Fig. 5.13, where the proton...
beam is directed to the microprobe beam line through a 90° bending magnet. To improve the detection resolution, the Si detector is cooled with nitrogen, since the photon energy is lower than the ion energy, the amount of electron/hole pair formed is lower, and therefore in PIXE, and in all techniques that detect X-rays, the detectors are cooled in order to improve their sensitivity. The X-ray detector consisted of a Si(Li) detector with 8 μm thick Be window and 145 eV resolution. The detector lies in the IBM geometry at an angle of 45° with a crystal active area of 70 mm². This detector has a solid angle of 112 msr, and is placed 25 mm from the sample, where the angle between the normal to the sample surface and the beam is 0°. In the experimental arrangement used for PIXE a Mylar 50 filter is placed between the sample and the detector such that the beam does not reach the X-ray detector. PIXE data was analyzed with Dan32 software [122].

This work was completed with the analysis of the four samples using the X-ray diffraction technique. The instrument used for this technique was a High-Temperature Double Crystal X-ray Diffractometer (Hotbird), as shown in Fig. 5.14, equipped with a copper anode source, with a double crystal monochromator, Ge (220), a scintillation detector and a Bruker goniometer with six axis. The diffractometer has seven important components, as shown in Fig. 5.15: (1) Scintillation detector; (2) 0.1 mm collimator; (3) Three axis goniometer with angular motion (ϕ,ω,χ), used to perform the scans, and three linear axes (x, y, z), that control the sample holder, and thus serve for the positioning of the sample relative to the beam; (4) 0.2 mm collimator; (5) Monochromator with two germanium crystals (220), that selects the wavelengths only of the $K_{α1}$ emission line of the copper, and limits the vertical divergence of the beam; (6) Göbel parabolic mirror to focus the beam and making it parallel and monochromatic; (7) X-ray source, with Cu anode. The software used for data analysis was Spectra Adjust [123].
RBS Experimental Results

RBS measurements were made with a 1940 keV +H beam with the detector positioned at an angle of 140° with the incident beam. Ho and Er were not observed due to their lower concentration in the sample, whereas Ir and Au are indistinguishable since their atomic mass is only ~ 5 amu apart, below the ~2 keV detector resolution. The NDF fit program provided the list of all the elements present in the sample. This simulation program takes into account the stopping power of the incident ion into the target material, calculated with SRIM. In this way, it is possible to separate the different contributions of the elements in the RBS spectrum, as shown in Fig. 5.16 and Fig. 5.17. The RBS analysis plot of holmium sample and the ones of the remaining samples are shown in different figures since, as previously noted, the analyzes were conducted at different times. For this reason, the holmium sample RBS plot can not be overlapped with the ones of the remaining samples.

![RBS spectrum of Ho implanted sample.](image-url)

Fig. 5.16: RBS spectrum of Ho implanted sample.
Comparing the implanted samples with the reference one (Au in the graph), a decrease in the Au layer thickness can be clearly seen from the energy difference.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Au thickness average (nm)</th>
<th>AuHo</th>
<th>AuEr</th>
<th>AuHoEr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Au thickness average</td>
<td>2305</td>
<td>898</td>
<td>1481</td>
<td>1032</td>
</tr>
<tr>
<td>Au thickness standard</td>
<td>0</td>
<td>351</td>
<td>286</td>
<td>373</td>
</tr>
</tbody>
</table>

Among the three implanted samples, the highest decrease in Au layer thickness occurred in the Ho and Er implanted sample and the lowest decrease can be seen in the Er implanted sample, as shown in Table 5.2. On the other hand, between the Si layer peak and the Au layer peak (~ 500 channel) there is a less abrupt transition in the implanted samples, suggesting diffusion of Au in Si, however with the Ir sticking layer between both layers, a more realistic interpretation would be the diffusion between these three elements.

To evaluate the sample thicknesses, the NDF fit program was used and the Au depth profile of the 4 samples is showed in Fig. 5.18. The results identify a 2.3 \( \mu \text{m} \) thickness for the reference sample, obtained from the depth value at 50% Au concentration value, consistent with the deposition estimation thickness of 2-3 \( \mu \text{m} \). Comparing all samples, the implanted samples show a Au film thickness decrease. The decrease was \(~39\%\) for the sample implanted with Er, \(~61\%\) for the sample implanted with Ho, and \(~65\%\) for the sample implanted with Ho and Er. This was an expected result, since the Ho+Er implanted sample was the one exposed to a longer implantation ion beam bombardment, increasing the Au surface loss due to the sputtering process, in accordance with the SRIM simulation suggesting that Au sputtering may occur as a consequence of the implantation parameters of this experiment doses on the order of \(10^{16} \text{ atm/cm}^2\) and an energy of 250 keV. The Au and Ir elements are not distinguishable in the RBS measurements, the values for the Au layer thickness depends therefore on the 70 nm thickness assumption for the Ir layer and the explicit incorporation of roughness in the Au surface using a gama-distribution on the fitting process. The results indicate that the fit with the highest Au thickness standard deviation is the
Ho+Er implanted sample (373 nm), then the Ho implanted sample (351 nm) and, at last, the Er implanted sample (286 nm), as in Table 5.2. All thickness values are calculated assuming the bulk density of the elements.

![PIXE spectra](image)

**Fig. 5.18**: Au concentration as a function of Au thickness for the 4 samples.

**PIXE Experimental Results**

The Dan32 fit program for PIXE spectra provided the information on the elements present in the samples, and the PIXE spectra of the four samples are shown in Fig. 5.19. The results indicate a reduction in the number of counts of the implanted samples compared to the reference sample, as expected from the RBS analysis.

![PIXE spectra](image)

**Fig. 5.19**: PIXE spectra of the four samples. The first peak in the spectra is a “null peak”. This peak is used to define the zero of the system and to verify the stability of the electronics.
The energy characteristic X-ray lines for each element are identified for each spectra line given by Dan32 fit program, and displayed in Fig. 5.20. The spectrum from Fig. 5.20 enables to identify not only the expected elements but also the impurities and contamination elements.

The implantation elements, Ho and Er, are clearly present even if with a low concentration value relative to the concentration value of Au. Dan32 fit program is able to match each line of the spectrum to an element. The peaks identification in Fig. 5.20 are shown in Table 5.3.

<table>
<thead>
<tr>
<th>Element</th>
<th>Line</th>
<th>Energy (keV)</th>
<th>Line Spectrum number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>$K_{\alpha_1}$</td>
<td>1.740</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>$M_{\alpha_1}/M_{\alpha_2}$</td>
<td>2.122/2.118</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>$M_{\gamma_1}/M_{\gamma_2}$</td>
<td>2.410/2.391</td>
<td>2</td>
</tr>
<tr>
<td>Au</td>
<td>$L_1$</td>
<td>8.493</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>$L_{\alpha_1}/L_{\alpha_2}$</td>
<td>9.713/9.628</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>$L_{\rho_1}/L_{\rho_2}$</td>
<td>11.442/11.585</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>$L_{\gamma_1}/L_{\gamma_2}$</td>
<td>13.381/13.626</td>
<td>6</td>
</tr>
<tr>
<td>Cr</td>
<td>$K_{\alpha_2}/K_{\alpha_1}$</td>
<td>5.406/5.415</td>
<td>22</td>
</tr>
<tr>
<td></td>
<td>$K_{\beta_1,3}$</td>
<td>5.947</td>
<td>23</td>
</tr>
<tr>
<td>Ho</td>
<td>$L_{\alpha_1}/L_{\alpha_2}$</td>
<td>6.680/6.720</td>
<td>24</td>
</tr>
<tr>
<td>Er</td>
<td>$L_{\alpha_1}/L_{\alpha_2}$</td>
<td>6.905/6.949</td>
<td>25</td>
</tr>
<tr>
<td>Fe</td>
<td>$K_{\alpha_2}/K_{\alpha_1}$</td>
<td>6.390/6.403</td>
<td>7</td>
</tr>
<tr>
<td>Ni</td>
<td>$K_{\alpha_2}/K_{\alpha_1}$</td>
<td>7.460/7.478</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>$K_{\beta_1,3}$</td>
<td>8.264</td>
<td>9</td>
</tr>
</tbody>
</table>

Table 5.3: Characteristic X-ray lines energy values of elements present in the Ho and Er implanted sample.

Peaks numbered from 1 to 19 are present in all 4 samples PIXE spectra. The ones that are not identified in Table 5.3 can be attributed to elements as ruthenium, titanium, and zirconium, since they are present during the experimental procedure of film production and analysis (sample holders...
made of stainless steel; Argon present in the chambers; etc). Peaks 14, 15 and 16 are attributed to Au and Ir from the samples, but are indistinguishable due to their close energy signatures.

The Si peak doesn’t appear in the reference sample spectrum. The implanted samples suffered, as indicated from the RBS study, a Au sputtering process, and therefore showing a decrease in the Au layer thickness in comparison with the reference sample. In these samples there is the possibility for the K X-Ray line characteristic of Si not be absorbed by the Au layer, since due to the Au layer thickness reduction there is the probability for the X-ray to pass through this layer and be detected.

The total concentration of each element for all samples is displayed in Fig. 5.21. This plot shows the difference in the concentration of each element present in each sample.

![Fig. 5.21: Total concentration of each element in the four samples.](image)

PIXE fit program indicated a Ho amount in Ho implanted sample of $1.1 \times 10^{15}$ at/cm$^2$, and in Ho+Er implanted sample an amount of $4.1 \times 10^{14}$ at/cm$^2$. The amount of Er present in the sample implanted with Er was $4.9 \times 10^{14}$ at/cm$^2$ and in the one implanted with Ho+Er was $7.9 \times 10^{14}$ at/cm$^2$. From these results the Ho discrepancy from the implantation parameters was $\sim 7.9 \times 10^{16}$ at/cm$^2$ (1.3% of the dose value) in the Ho implanted sample and $\sim 2.4 \times 10^{16}$ at/cm$^2$ (1.7% of the dose value) in the sample implanted with Ho+Er. The Er loss was $\sim 5.7 \times 10^{16}$ at/cm$^2$ (0.9% of the dose value) in the sample implanted with Ho and $\sim 5.6 \times 10^{16}$ at/cm$^2$ (1.4% of the dose value) in the Ho+Er implanted sample. Cr seems also to be present with the same concentration in all the implanted samples, but not in the reference sample, indicating that this element was possibly introduced during the implantation process. Fe and Ni are small impurities present in the reference sample, before any implantation.

**XRD Experimental Results**

X-ray Diffraction spectra, shown in Fig. 5.22, were performed with oblique geometry, an angle of $2^\circ$ with respect to the sample surface.
Analyzing this figure, it is possible to conclude that the peaks from the X-ray diffraction spectrum are due to a face centered crystalline structure, since the allowed reflections for this type of structure obey the condition \( hkl \) all odd or all even, as shown in Table 5.4.

Comparing this experimental spectrum with standard reference patterns, one can conclude that only the top Au film layer was measured and only part of the peaks characteristic of this element were seen. The peaks identification in the spectra of all samples indicates a good match with the ones from the standard pattern of Au. The major difference between the experimental spectra and the standard spectrum relies on the number of counts. Compared to the reference sample, the implanted samples have a decrease in the number of counts for all the peaks. To further understand the impact of the implantation process, a simple analysis of the interplanar distance, \( d \) (decrease in \( 2\theta \)), was conducted and the results are shown in Table 5.5.

### Table 5.4: X-ray diffraction spectrum analysis of reference sample.

<table>
<thead>
<tr>
<th>Peak #</th>
<th>( 2\theta ) ((^\circ))</th>
<th>( \theta ) ((^\circ))</th>
<th>( d ) (Å)</th>
<th>( \sqrt{h^2 + k^2 + l^2} )</th>
<th>( h^2 + k^2 + l^2 )</th>
<th>( h )</th>
<th>( k )</th>
<th>( l )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>38.28</td>
<td>19.14</td>
<td>2.3494</td>
<td>1.7366</td>
<td>3</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>44.50</td>
<td>22.25</td>
<td>2.0343</td>
<td>2.0056</td>
<td>4</td>
<td>2</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>64.71</td>
<td>32.36</td>
<td>1.4392</td>
<td>2.8350</td>
<td>8</td>
<td>2</td>
<td>2</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>77.72</td>
<td>38.86</td>
<td>1.2277</td>
<td>3.3232</td>
<td>11</td>
<td>3</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>5</td>
<td>81.86</td>
<td>40.93</td>
<td>1.1758</td>
<td>3.4700</td>
<td>12</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
</tbody>
</table>

### Table 5.5: Interplanar distance of each peak in each sample.

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Au</th>
<th>AuHo</th>
<th>AuEr</th>
<th>AuHoEr</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.3494</td>
<td>2.3564</td>
<td>2.3527</td>
<td>2.3530</td>
</tr>
<tr>
<td>2</td>
<td>2.0343</td>
<td>2.0389</td>
<td>2.0372</td>
<td>2.0376</td>
</tr>
<tr>
<td>3</td>
<td>1.4392</td>
<td>1.4413</td>
<td>1.4403</td>
<td>1.4404</td>
</tr>
<tr>
<td>4</td>
<td>1.2277</td>
<td>1.2293</td>
<td>1.2289</td>
<td>1.2287</td>
</tr>
<tr>
<td>5</td>
<td>1.1758</td>
<td>1.1767</td>
<td>1.1767</td>
<td>1.1766</td>
</tr>
</tbody>
</table>

Fig. 5.22: X-ray diffraction experimental spectra. The long tail for \( 2\theta < 25^\circ \) is a nonzero starting point that has no physical meaning, since it corresponds to instrumental background signal.
A possible interpretation is that when ions are bombarded into the sample, the implanted ones, cause the distance between the sample ions to increase, in this case, the distance between Au ions was increased. Due to Bragg's law, an increase in the lattice space parameter \( d \) means a \( \theta \) decrease. The results of the interplanar distance dependence with the implantation parameters, as the final dose of Au, Ho and Er in the samples indicate that there is some change since the interatomic distance increased after implantation. Although there was an increase in the interatomic distance after the implantation process, there seems to be no correlation with the dose of Ho and Er implanted (10.6 and 4.1 \( \text{E}14 \) at/cm\(^2\) of Ho final dose, and 4.9 and 7.9 \( \text{E}14 \) at/cm\(^2\) of Er final dose, for Ho and Ho+Er implanted samples, respectively), nor with the implantation time (154, 304, 535 min for Ho, Er, and Ho+Er implanted samples, respectively). Also, there is no correlation to the global amount of Au lost after implantation from RBS technique (61, 39, 65 % of Au lost for Ho, Er, and Ho+Er implanted samples, respectively).

Discussion and Conclusions

The ion implantation process of non-radioactive metallic Ho and Er onto thin Au films was studied using RBS, PIXE and X-ray techniques, and allowed to further understand the properties change due to the implantation experimental parameters.

RBS technique indicated that a diffusion process may have occurred between Au, Ir and Si, and furthermore a decrease in the Au film layer thickness during implantation. Au loss could be seen by comparing the implanted samples and reference sample spectra. The sample with the highest Au thickness decrease after implantation was the Ho+Er sample (65% loss) attributed to a higher beam exposure time. PIXE technique provided the effective concentration values of implanted Ho and Er, and also allowed to identify other elements beside Au and Ir present in the sample from contaminations and impurities. A way to overcome the contamination problem will be, for example, to place the samples in a sample holder of ceramic or on a Si wafer, where the sputtering coefficient is very low, and thus, impurities such as Fe, Ni and Cr will no longer contaminate the sample. X-ray diffraction technique results on the Au surface layer of the samples, indicated that the crystalline structure was not damaged, and a possible increase in the interplanar distance of the implanted samples. These results also showed a decrease in the number of counts for implanted samples, suggesting a possible degradation of the surface Au film.

Complementary RBS and PIXE techniques provided information on the presence of Si K-line in the implanted samples showing a higher intensity in the Ho+Er implanted sample. This line is not present in the sample implanted with Er or in the reference sample, suggesting that the decrease in the Au thickness due to sputtering triggered the X-rays absorption from the Si layer, resulting in a smaller number of detected events.

A similar experiment had already been performed [44] and the parameters originally set for implantation have been fully achieved in this previous experiment in 12 samples, with no sample damage nor sputtering nor contamination processes. Comparing both experiments, the following differences can be found between the experimental setting parameters and now clearly identified: (1) beam exposure time of the sample, and longer exposure times the higher the amount of gold removed; (2) higher beam current, leading to a high decrease in Au thickness and to sputtering processes; and (3) higher power density, increasing the Au sputtering. Even though it was not possible to achieve the implantation parameters as planned guaranteeing the high quality of the thin films, this experiment enabled to further understand the implantation of Er and Ho process in Au thin films and conclude that a special care on the implantation process must be taken to in order to optimize the detector absorber performance for HOLMES, and thus proceed with the measure the electron neutrino mass.
6 Low Temperature Measurements

In a TES detector, the incident particle breaks Cooper pairs, altering the measured resistance of the sensor. The TES superconducting film and substrate are cooled below the film transition temperature in a cryostat, and the film is then biased back up into its transition. In this state, the film acts as a particle detector, since a small amount of deposited energy in the absorber will produce a large resistance change. The basic thermal detector model is a heat capacity linked to a cooling bath by a thermal conductance, as in Fig. 6.1

The film is wired into a bias circuit and raised into its transition region with an electrical current. If the voltage across the sensor is held approximately constant, the bias point is stable. If the detector absorbs energy from an incoming particle, both its temperature and resistance will rise. This means that the Joule heating, $P_{\text{joule}} = V^2/R$, actually decreases, pushing the detector back towards its initial bias temperature. Conversely, if cryostat fluctuations cause the detector temperature to drop, the Joule heating actually increases, again pushing the detector back towards its original bias point.

The success of a voltage-biasing scheme relies upon the thermal link between the film and the rest of the system, as already explained in Chapter 3. If the link is too strong, heating the sensor will simply heat the entire substrate, detector stage, and cryostat. The cryostat cools the substrate, which cools the film phonon system, which cools the film electron system. TESs are always voltage-biased, and their behavior can be understood in terms of small deviations from a steady-state balance between the Joule heating due to the bias current and the cooling power of the substrate.

Detection and amplification of a signal from any detector requires impedance-matching the amplifier with the device. Since TESs are low-impedance, to detect the resistance change of the TES due to an incident photon, this device is inductively coupled to a Superconducting QUantum Interference Device (SQUID). A SQUID is a very sensitive magnetic flux to voltage converter. It consists of a superconducting loop with two Josephson junctions in parallel. Looking at the SQUID feedback signal gives us a direct window (with some gain factors) onto the current in the TES.

In HOLMES experiment, TES using SQUIDs for the electronic readout are the best candidates, since SQUIDs can be easily impedance-matched to low-resistance TES detectors, few Ohms, and it is also possible to multiplex the readout of TES detectors using SQUID amplifiers [125].

In this experiment, the critical temperature of a Ti/Au/Ti 3 layer TES sensor is measured using a SQUID current amplifier. This procedure was useful to familiarize myself with all the variables that are involved in the characterization and subsequent measurement of an event through a TES detector. Regardless, the fact of a bolometer and microcalorimeter measure different quantities, as explained in Chapter 3, the procedure for its characterization is the same.
6.1 Experimental Setup

In order to measure superconductive to normal transition curve of a sensor TES bolometer, this sensor was coupled to a supracon SQUID Model CS-blue, with a Nb/Al - AlOx/Nb junction and a working temperature between 1 and 5 K. This device is a low-$T_c$ dc SQUID designed as a current sensor, and is suited as a preamplifier of current signals with small amplitudes, e.g., signals of transition edge sensors, TES. This device operates essentially as a current-voltage converter extremely sensitive, in which the current that arrives at its terminals comes from interaction of the sample with the detection coils. The result of this interaction is the induction current in these coils. The detection coils (made of superconducting material) are connected to a superconducting coil to the SQUID input through bonding wires, also superconducting, constituting a closed circuit. As a result, given the highly linear response of the SQUID, these variations in current produce variations in the output voltage, which is proportional to the magnetic moment of the sample. It is this linear response that allows magnetic measurements with SQUID being three orders of magnitude higher than with previous devices. It is therefore possible to measure the intensity of the magnetic field and the current that created it. The power supply circuit and read-out for the SQUID measurements is shown in Fig. 6.2. Due to the high sensitivity of this device, this device can detect (undesirably) small fluctuations of the superconducting coil applied field and also of the field existing in the laboratory. Thus, it is important that the SQUID is shielded, in some way, from these fluctuations.

To achieve the SQUID working temperature, the device is placed inside a Kelvinox 25 dilution refrigerator (cryostat) from Oxford Instruments, at Istituto Nazionale di Fisica Nucleare in Genoa, as shown in Fig. 6.5a, shielded by a magnetic shield and a Faraday Cage, capable of performing -60 dB at 2 GHz.

![Fig. 6.2: Diagram of the circuit for SQUID measurements. The power supply for TES circuit is at left, and the SQUID circuit is at right (42).](image-url)
6.2 Dilution Refrigerator

A cryostat is a device used to maintain low cryogenic temperatures of samples or devices mounted within the cryostat. In this work the refrigeration method used was a cryogenic fluid bath such as liquid helium. A $^3\text{He}/^4\text{He}$ dilution refrigerator is a cryogenic device that provides continuous cooling to temperatures as low as 2 mK, where $^3\text{He}$ is the working fluid, which is circulated by vacuum pumps at room temperature. The cooling power of the refrigerator used can achieve more than 400 mW, which is provided by the heat of mixing of the $^3\text{He}$ and $^4\text{He}$ isotopes. When a mixture of $^3\text{He}$ and $^4\text{He}$ is cooled below 870 mK, it separates into two phases, as shown in Fig. 6.3. The lighter "concentrated phase" is rich in $^3\text{He}$ and the heavier "dilute phase" is rich in $^4\text{He}$, containing about 6.6% $^3\text{He}$ and 93.4% $^4\text{He}$. The concentration of $^3\text{He}$ in each phase depends upon the temperature. Since the enthalpy of the $^3\text{He}$ in the two phases is different, it is possible to obtain cooling by evaporating the $^3\text{He}$ from the concentrated phase into the dilute phase (endothermic process, removes heat from the mixing chamber environment). The concentrated and dilute phases separate and a phase boundary is established in the mixing chamber, where the cooling process takes place. To establish continuous cooling one must promote the flow of $^3\text{He}$ across the phase boundary in a continuous process. This is achieved by raising the temperature of the dilute phase to ~700 mK outside of the mixing chamber in the still. The vapour pressure of $^3\text{He}$ at this temperature is two orders of magnitude higher than that of $^4\text{He}$ allowing $^4\text{He}$ to be preferentially pumped using external room temperature mechanical pumps. This exhausted $^3\text{He}$ can be returned to the system, entering the cryostat at a pressure of a few hundred millibar and being precooled and purified by liquid nitrogen at 77 K and a $^4\text{He}$ bath at 4.2 K. After this, $^3\text{He}$ enters to the vacuum chamber where is further cooled to a temperature of ~1 K in a pumped $^4\text{He}$ bath, called the "1 K pot", as shown in Fig. 6.4. In the 1 K pot the pressure is decreased (depressing the boiling point) and the condensation heat is removed, condensing the $^3\text{He}$ gas. Finally, at the still, the $^3\text{He}$ flows through superfluid $^4\text{He}$ which is at rest. The pressure in the still is kept low (about 10 Pa) by the pumps at room temperature. The vapor in the still is practically pure $^3\text{He}$, which has a much higher partial pressure than $^4\text{He}$ at 500-700 mK. The pump therefore creates an osmotic pressure difference, which drives more $^3\text{He}$ from the concentrated to dilute phases in the mixing chamber, and then up from the mixing chamber to the still. Heat is supplied to the still to maintain a steady flow of $^3\text{He}$. The pumps compress then $^3\text{He}$ to a pressure of a few hundred millibar and feed it

Fig. 6.3: Phase diagram of liquid $^3\text{He}/^4\text{He}$ mixtures showing the phase separation [126].

Fig. 6.4: Dilution refrigerator scheme [127].
back into the cryostat, completing the cycle. When leaving the still to its way down, \(^3\)He is further cooled through heat exchange with the exiting stream using a continuous heat exchanger \(\sim 150\) mK and a series of silver sinter step heat exchangers from 100 mk to 20 mK, before being reintroduced to the mixing chamber to continue the process.

Thermally fixed to the mixing chamber is located a oxygen-free high-conductivity, OFHC, copper support with high diffusivity, called the "block". The block is connected to the mixing chamber with a calibrated conductance to filter the thermal fluctuations of the mixing chamber in order to reach the highest thermal stability. In the cooling process of this experimental work, the temperature was controlled by the thermometers present in the block, mixing chamber and in the 1K pot, and the pressure controlled in the still, condensation line, 1K pot and in the cylinder, where the mixture is recovered at the end of the operation. The only calibrated thermometer was the one present in the block, calibrated by a reference thermistor doped with germanium.

To protect the cooling platform from heating, the dilution unit and the 1 K pot are housed in a vacuum with a radiation shield from either the still or 100 mK cold plate that surrounds the heat exchangers and sample space below the mixing chamber.

Gaining access to the sample is made by removing the insert from the cryostat, warming it to room temperature and removing the inner vacuum chamber (IVC) tail to gain access to the mixing chamber base.

Fig. 6.5: (a) Kelvinox 25 dilution refrigerator from Oxford Instruments used at Istituto Nazionale di Fisica Nucleare in Genoa; (b) TES Spiderweb Bolometer inside the dilution refrigerator; (c) SQUID Model CS-blue inside the dilution refrigerator [128].
Inside the cryostat, the SQUID is placed on the other side of the copper sample holder, attached to the top of the IVC, as shown in Fig. 6.5c, which is at 4.2 K. For this reason the circuit connecting wires are superconducting, thereby contributing to a low thermal conductance value, in which otherwise, the sample holder would not reach the working temperature. Inside the cryostat was installed a connector to supply power to a single detector, in order to measure the SQUID response.

In the SQUID characterization phase a function generator is used, feeding the circuit with a triangular wave at low frequency. The signal is filtered by a low pass RC circuit with a cutoff frequency of 1 kHz, before entering the cabin that protects the cryostat by means of radiofrequency. The output voltage is proportional to the current $I_x$ flowing through the sample [42]:

$$V_{out} = G \cdot I_x \tag{6.1}$$

where $G$ is the SQUID transresistance.

The voltage at the terminals of the sample is given by the ratio [42]:

$$V_x = \frac{R_s R_x}{(R_s + R_x) R_L}, \quad V_{in} = \frac{R_s}{R_L}, V_{in} - R_s I_x \tag{6.2}$$

where $R_s = V_x / I_x$ and the term in the denominator was neglected, since $(R_s + R_x) \ll R_L$, since $R_s$ and $R_x$ are of the order of tenths of mΩ, while $R_s$ is of the order of kΩ.

In the limit where the sample is completely transit, that is, $R_s \ll R_x$, Eq. 6.2 becomes [42]:

$$V_x = V_{in} \cdot \frac{R_s}{R_L} \tag{6.3}$$

From $V_{in}$ and $V_{out}$ measurements, it is possible to obtain the thermal and electrical characteristics of the sample, the resistance as a function of the temperature, the current flowing through the sample, and also the dissipated power as a function of the polarization.

The sample used was a TES Spiderweb Bolometer for Multi-mode Cavity Microwave Detect [129], Ti/Au/Ti 3 layer TES sensor with $T_c$ tuned in the 330-380 mK and 2 mK transition width, and is shown in Fig. 6.5b. Before starting the measurement with the SQUID the thermometer in the block was tested and the offset was stable over time. Since this thermometer is a thermistor the temperature values were read in resistance units, ohms.

The load resistance value, $R_L$, as in Fig. 6.2, can vary between two values chosen by the user: 47 kΩ or 4.66 kΩ. The highest value for the load resistance was used for this measurements, since for the lowest value the amplitude of the signal was very high. Using a triangular wave for the input signal, one obtain $V_{out}(V_{in})$. Since, the TES is superconductor at low temperature, when it is in the superconducting state the resistance is zero, so in the circuit, shown in Fig. 20, the only resistance value is due to $R_s$ in the loop. When the TES is in the metallic state the resistance of the loop is greater than $R_s$, it is $R_s + R_{TES}$.

To calculate the current-voltage conversion value, $G$, first one needs to set the frequency value for the input signal necessary to obtain the characteristic SQUID signal. This frequency value is given by the SQUID manual, and for the SQUID used this interval is 50-100 Hz. The value chosen was 100 Hz. For this frequency value the period of the output signal was measured and given to the input signal, in order to see the SQUID response. With this procedure is possible to know which voltage one needs to give to the SQUID device, in order to obtain a flux quantum. To reduce the noise the signal average was calculated and a value of 49.6 mV was obtained for the output voltage (peak to peak). From the SQUID datasheet one finds the current needed to obtain a flux quantum, which in this case was $0.21 \, \mu A/\varphi_0$. So, the conversion factor for the SQUID device used was:

$$G = \frac{49.6 \, mV/\varphi_0}{0.21 \, \mu A/\varphi_0} = 236.2 \times 10^3 \, V/A$$
This value was measured with an associated error of 10%.

To obtain the resistance-temperature curve of the TES sensor the temperature was controlled, varying in a range of 38 mK with steps of approximately 2 mK. The input and output signals of the system were acquired, for each chosen temperature value. To calculate the value of the current and of the voltage in the TES Eqs. 6.1 and 6.3 were used. The resistance value of the TES for each temperature was calculated plotting the current in the TES \( I_x \) as a function of the voltage in the TES \( V_x \). The plot were interpolated with a straight line, \( y = ax + b \), and the slope value, \( a \), was taken and used to calculate the resistance of the TES as:

\[
R_x = \frac{1}{a} \tag{6.4}
\]

Using this procedure the superconductor to normal state transition curve of the TES was obtained, by plotting the resistance of the TES, \( R_x \), as a function of the temperature.

### 6.3 Electronics and Signal Readout

The SQUID output signal as a function of the input signal is showed in Fig. 6.6, for a frequency of 100 Hz and an input voltage of 10 V. The output vs input signal was measured for a temperature range between 322 mK and 358 mK.

![Graph of output vs input signal](image)

**Fig. 6.6:** Output vs input signal for a temperature range between 322 and 358 mK (160 and 130 Ω), a frequency of 100 Hz and a input voltage of 10 V.
This I-V characteristic measured, in Fig. 6.6, corresponds approximately to what one should get when in the circuit there is some additional resistance in series with the TES, which is the case of the circuit used, as shown in Fig. 6.2. The TES should show an electro-thermal feedback with a reciprocal part of I-V (negative resistance) if it is biased with voltage source. This characteristic can also be seen from Fig. 6.6.

![Image of Resistance vs Temperature curve of TES sample](resistance_vs_temp.png)

**Fig. 6.7:** Resistance vs Temperature curve of TES sample, for a frequency of 100 Hz and a input voltage of 10 V.

This plot enables the characterization of the TES sensor. It shows a typical resistance vs temperature curve of a superconducting film. Fig. 6.7 shows that the resistance of the sensor changes from 345 mK to 348.7 mK. This is the transition region of the TES. In the range of 322.4 to 345 mK is the superconductor region of the TES, since it is a region of very low resistance, 0.04 Ω. Above 348.7 mK the TES is at the normal state with a resistance value above 0.1 Ω. Since the sample has nonzero resistance at the contacts between the superconductor and its metal leads, the lowest temperature resistance won't be zero.

6.4 Discussion and Conclusions

The Ti/Au/Ti 3 layer TES sensor was designed to have the following characteristics: a critical temperature tuned in the 330-380 mK and a narrow transition width. The results showed a transition width of 3.73 mK, between 345 mK and 348.7 mK, which is close to the value for which this sample was designed.
This TES sample would not be suitable for MARE or HOLMES experiments since the critical temperature value of 345 mK, is much higher than the one required for both experiments (~80 m). However, the detector and sensor technology, and experimental procedure used is equivalent for the range of temperature required for a measurement of a Ir/Au or Mo/Au bilayers TES sensor.
7 Conclusions and Perspectives

The experiment goal is the microcalorimetric measurement of the electron neutrino mass with a $^{163}$Ho source embedded into the absorber. Knowing the thermal properties at the working temperature of the microcalorimeter components is critical for choosing the absorber material and for optimizing detector performance. This experiment has as potential absorber material gold due to its high conductivity.

For this work, results from Ho and Er isotopes implantation into gold thin films were taken in order to improve the parameters needed when a concentration of one order of magnitude higher than the one used in a previous experiment [44] of these elements is implanted into the detector absorber. These needs to be fully optimize, since as higher the concentration of holmium radioactive in the absorber, higher will be the number of the decays, and higher will be the statistics in the region close to the end-point of the EC spectrum. Also, knowing the thermal properties at the working temperature of microcalorimeters is critical for choosing the absorber material and for optimizing the detector performance. In particular, it is important to understand if implanting the radioactive material in gold changes its heat capacity. The previous experiment used a bolometric technique to measure the heat capacity of gold films implanted with various concentrations of holmium and erbium, in the temperature range 70 mK to 300 mK. Their results showed that the specific heat capacity of the gold films is not affected by the implantation of Ho and Er, making this a viable option for a future microcalorimeter holmium experiment. This experiment used dose values of Ho and Er of $\sim 10^{15}$ at/cm$^2$, and the implantation results showed that the implantation parameters were achieved without gold sputtering in the samples. Increasing one order of magnitude the dose value, $10^{16}$ at/cm$^2$, results in a different outcoming, since in this case the results showed that a gold amount of almost 50% was sputtered from the samples and also only 5% or less of Ho and Er dose value was implanted in the samples. From these results it is important in the future, to know the phenomena that led to these results in order to improve the implantation process of doses of Ho and Er even higher, and proceed to the heat capacity measurements to conclude whether this property changes with these dose higher values.

A TES Bolometer device was readout using a SQUID, and the critical temperature of the 3 layer sensor was measured.

Microcalorimeters with gold film as absorber and energy resolution better than 2 eV already exist. The next step is to replace the gold film absorber with the $^{163}$Ho implanted absorber in such microcalorimeters. This modified microcalorimeter with a source embedded absorber can be used to carry out a small size, short-time scale pilot experiment for testing the potential of a Holmium experiment.
References


[84] Low Temperature Detectors; Principles and Applications. LTD 13, 2009.
[128] Instituto Nazionale di FisicaNucleare, sezione di Genova, INFN. Available at: http://www.ge.infn.it/