

Detection of Second Sound in He-II for Thermal Quench Mapping of Superconducting Radio Frequency Accelerating Cavities

Master Thesis of

cand. chem.-Ing. Tobias Stegmaier, B.Sc.

At the Department of Chemical and Process Engineering



Institut for Technical Thermodynamics
and Refrigeration



European Organisation for Nuclear
Research

Assignment:	Prof. Dr.-Ing. Steffen Grohmann (KIT, ITTK)
Second reviewer:	Prof. Dr.-Ing. Matthias Kind (KIT, TVT)
Supervisor:	Dr. Hernán Furci (CERN, BE-RF-SRF)
Second advisor:	Dr. Torsten Koettig (CERN, TE-CRG-CI)
Third advisor:	Dipl.-Ing. Benedikt Peters (KIT, ITEP)

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Thesis Topic

Institute of Technical Thermodynamics and Refrigeration (ITTK)

Head: Prof. Dr. habil. rer. nat. Sabine Enders

Engler-Bunte-Ring 21
76131 Karlsruhe, Germany

Phone: +49 721-608-4-2332

Fax: +49 721-608-4-2335

E-Mail: steffen.grohmann@kit.edu

Web: www.ttk.kit.edu

Official in charge: Prof. Dr.-Ing. Steffen Grohmann

Our reference:

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Detection of second sound in He-II for thermal quench mapping of superconducting radio frequency accelerating cavities

Deutscher Titel: Detektion des Zweiten Schalls in He-II zur thermischen Quenchkartierung von supraleitenden Hochfrequenz-Beschleunigerkavitäten

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Focus: ■ Literature study ■ Theory ■ Design ■ Experiment

Supervision: Dr. Hernan Furci (CERN, BE-RF-SRF, hernan.furci@cern.ch)
Dr. Torsten Koettig (CERN, TE-CRG-CI, torsten.koettig@cern.ch)
Dipl.-Ing. Benedikt Peters (KIT, ITP, benedikt.peters@kit.edu)

Assignment: Prof. Dr.-Ing. Steffen Grohmann

Scope of Work

Future projects for particle accelerators ask for increased performance of superconducting radio frequency (SRF) cavities by enhancement of the overall efficiency, in particular the reduction of dynamic losses at significant accelerating gradients. The ultimate performance limit is the quench of SRF cavities, i.e. the loss of superconductivity on a local hotspot. Such hotspots are caused by material/fabrication defects that lead to local field line concentrations.

The hotspots and the defects can be localised during cryogenic testing, using oscillating superleak transducers (OST). A network of OSTs is placed around the cavity and the quench location is calculated from the time shift of second sound signals using triangulation.

In a new study, the OSTs shall be replaced by newly developed transition edge sensors (TES), which provide higher spatial resolution and a response intensity linear with the heat flux. The TES is a thin-film thermometer. Its electric resistance varies steeply within the superconducting-to-normal transition, which makes it very sensitive to small temperature changes. The sensitive transition range, however, is limited to about 200 mK, but this range can be shifted along the temperature scale by the bias current.

The upgrade and development of a thermal mapping system based on TES requires a series of studies to be carried out in the frame of this thesis, including:

- Characterisation of the TES response in terms of distance, angle, intensity and time, using heat pulses in cryogenic tests with saturated HeII
- Comparison of different prototype TES with different film thicknesses
- Application of the characterised TES for thermal quench mapping of SRF cavities and comparison to the established OST method

The master thesis shall be written in English language. A two-sided summary in German shall be submitted together with the thesis. In addition, the results shall be presented in a 20 minutes presentation in the ITTK Institute seminar at KIT.

Name of Student: Tobias Stegmaier

Start of Work: 01.06.2017

A handwritten signature in blue ink, appearing to be 'S. Grohmann', written over a horizontal dashed line.

07.04.2017, Prof. Dr.-Ing. S. Grohmann

Declaration of own Work

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(Tobias Stegmaier)

Abstract

The development of future particle accelerators requires intensive testing of superconducting radio frequency cavities with different sizes and geometries. Non-contact thermometry quench localisation techniques proved to be beneficial for the localisation of surface defects that can originate a quench (sudden loss of superconducting state). These techniques are based on the detection of second sound in helium II.

Transition Edge Sensors (TES) are highly sensitive thin film thermometers with fast time response. In the present work, their capability as a thermal quench mapping device for superconducting radio frequency cavities is proven experimentally by detecting second sound waves emitted by SMD heaters in a He-II bath at saturated vapour pressure. A characterisation of the sensors at steady bath temperatures was conducted to calculate the thermal sensitivity. An intense metallurgical study of gold-tin TES with different compositions revealed important relations between the superconducting behaviour and the microstructure of the thin film. It was successfully demonstrated that heat treatment can be used to enhance the superconductivity of the sensors after the manufacturing process if desired. A strategy for optimal biasing of the sensors was developed to maximise the signal-to-noise-ratio of the measured second sound signal. The velocity of second sound at 1.8 K could be confirmed and the position of the heaters was successfully localised by trilateration with more than 90 % of success. In a dedicated testing setup, the three-dimensional propagation of second sound could be investigated in terms of angle and distance between heater and sensor. Especially the influence of the heater size and the detection angle could be studied, which reveals useful information for quench detection tests. Finally, the different nature of TES and Oscillating Superleak Transmitters (OST), another common second sound detector, was investigated experimentally showing that the temperature measurement of the TES is less angle dependent than the detection of the movement of the two components in helium II by the OST.

Thesis summary in German

Im Laufe der letzten Jahrzehnte haben sich supraleitende Hohlraumresonatoren, auch Hochfrequenzkavitäten genannt, als wichtiges Schlüsselement auf dem Feld der Teilchen- und Hochenergiephysik etabliert. Mittels eines oszillierenden elektromagnetischen Feldes erhöhen sie die Energie von Teilchen in Elementarteilchenbeschleunigern wie beispielsweise dem Large Hadron Collider (LHC) am CERN.

Der Bedarf nach immer leistungstärkeren Kavitäten erfordert eine ausgiebige Überprüfung der Resonatoren zur Qualitätssicherung, bevor sie in den eigentlichen Beschleuniger eingebaut werden können. Besonders Material- und Oberflächendefekte mit einer Größe im Mikrometerbereich, die während des Herstellungsprozesses entstehen, können die maximale Leistung der Kavität erheblich herabsetzen. Mit zunehmender Intensität des elektromagnetischen Feldes innerhalb der Kavität heizen sich diese Defekte auf und führen schlussendlich zu einem verfrühten Verlust der Supraleitung, einem sogenannten Quench, welcher die Leistungsgrenze einer Kavität darstellt. Deswegen ist es unerlässlich, diese Schwachstellen zu lokalisieren, um sie korrigieren und ihren Ursprung verstehen zu können. Dies wird in der Regel mit einer Vielzahl an Temperatursensoren realisiert, die nahe oder auf der Oberfläche der Kavität angebracht werden und den Außentemperaturverlauf des Resonators während des Testsvorgang im flüssigen Helium Bad aufzeichnen. Diese Methode ist jedoch sehr aufwendig und teuer, da die Messausrüstung je nach Resonator unterschiedlichen Formen individuell angepasst werden muss.

Ein flexibler Ansatz zur Quench Lokalisierung wurde 2008 mit den sogenannten Oscillating Superleak Transducers (OST) eingeführt. OST sind in der Lage, Temperaturwellen in suprafluiden Helium zu detektieren. Diese Temperaturwellen, besser bekannt als zweiter Schall, werden von einer Wärmequelle ausgesendet und bewegen sich mit konstanter Geschwindigkeit fort. Durch das Messen der Laufzeit des zweiten Schalls mit mindestens drei Sensoren kann der genaue Ursprung der Welle und somit der Defekt durch Trilateration lokalisiert werden. Ein großer Nachteil der OST ist jedoch, dass sie nicht die Temperaturvariation des zweiten Schalls messen können.

Darum wurde 2016 am CERN ein Projekt gestartet, bei dem sogenannte Transition Edge Sensors (TES) zur Quench Detektierung verwendet werden. TES sind Dünnschicht-Filmthermometer mit ausgezeichneter Reaktionszeit, die sich aufgrund ihrer materiellen Zusammensetzung im Übergang vom supra- zum normalleitenden Zustand befinden. Kleine Temperaturerhöhungen führen bereits zu einem großen Anstieg des elektrischen Widerstandes, was wiederum in einer hohen thermischen Sensitivität der TES in einem bestimmten Temperaturintervall resultiert. Dieser Bereich kann durch gezieltes treiben eines elektrischen Stroms zu höheren Temperaturen hin verschoben werden.

In der vorliegenden Arbeit werden Prototypen von Gold-Zinn TES mit unterschiedlicher Zusammensetzung charakterisiert. Intensive metallurgische Untersuchungen konnten Zu-

sammenhänge zwischen der Mikrostruktur und dem supraleitenden Verhalten der Sensoren aufzeigen. Diese Informationen können eine Grundlage zur Optimierung und Anpassung des Herstellungsprozesses der TES an gewünschte Rahmenbedingen wie Temperaturmessbereich bieten.

Durch das Verbessern der Supraleitung eines TES mittels Warmglühen bei 120 °C konnte erfolgreich gezeigt werden, dass TES mit unzureichender Supraleitung aufgrund suboptimaler Zusammensetzung nachträglich korrigiert werden können.

TES mit einer Zusammensetzung aus 20 nm Gold und 225 nm Zinn konnten als ausgezeichnete Detektoren für zweiten Schall im klassischen Temperaturbereich von supraleitenden Hochfrequenzkavitäten-Tests validiert werden. Dazu wurde ein Quench mittels kleiner Widerstandsheizter in einem suprafluiden Heliumbad simuliert. Die ermittelte Geschwindigkeit des zweiten Schalls bei 1.8 K liegt in guter Übereinstimmung mit Literaturwerten. Mit Hilfe eines simplen Trilaterationalgorithmus konnten des weiteren die genaue Position der verschiedenen Widerstandsheizter mit Millimeter-Präzision festgestellt werden.

Im letzten Abschnitt der hier vorgelegten Arbeit wird die Verbreitung des zweiten Schalls im dreidimensionalen Raum näher untersucht. In einem eigens dafür entwickelten Messstand konnte experimentell die Abhängigkeit der gemessenen Amplitude der Temperaturwelle vom Detektierungswinkel bewiesen werden. Auch ein Einfluss der Oberflächengröße der Wärmequelle konnte nachgewiesen werden. Diese Beobachtungen liefern wichtige Informationen über Effekte, die für spätere Analysen von quenchenden Hohlraumkavitäten unerlässlich sind. Abschließend beweist ein Vergleich zwischen einer TES und einer OST Messung den Unterschied in der Funktionsweise der beiden Detektoren und untermauert die Fähigkeit des TES, im Gegensatz zu den OST wirkliche Temperaturfluktuationen messen zu können.

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Symbols and Abbreviations used

Latin letters

Symbol	Description	Unit, value of constant
A	area	m^2
A_S	parameter	
B	magnetic flux density	T
B_c	critical magnetic flux density	T
C	heat capacity	$\text{Jkg}^{-1}\text{K}^{-1}$
c_p	heat capacity at constant pressure	$\text{Jkg}^{-1}\text{K}^{-1}$
c_v	heat capacity at constant volume	$\text{Jkg}^{-1}\text{K}^{-1}$
D	electric displacement field	Cm^{-2}
d	distance	m
d_m	actual known distance	m
d_{2s}	distance measured by time of flight	m
E	electric field	Vm^{-1}
E_0	zero-point energy	J
h	Planck constant	$6.626 \times 10^{-34} \text{ J s}$
I	electric current	A
J	electric current density	Am^{-2}
J_c	critical electric current density	Am^{-2}
j_b	momentum density of He-II	$\text{kgm}^{-2}\text{s}^{-1}$
k_B	Boltzmann constant	$1.380 \times 10^{-23} \text{ JK}^{-1}$
m	mass	kg
m_{2s}	slope of peak of second sound signal	Vs^{-1}
N_A	Avogadro constant	$6.022 \times 10^{23} \text{ mol}^{-1}$
n	exponent	
n_{Au}	amount of substance (gold)	mol
n_{Sn}	amount of substance (tin)	mol
P	power	W
p	pressure	Pa
Q	heat supply	W
$\dot{q}$	heat flux	Wm^{-2}
R	resistance	Ω
R_0	residual resistance	Ω
r	radius	m
s_b	specific entropy of He-II	$\text{Jkg}^{-1}\text{K}^{-1}$
s_n	specific entropy of normal fluid component	$\text{Jkg}^{-1}\text{K}^{-1}$
s_s	specific entropy of superfluid component	$\text{Jkg}^{-1}\text{K}^{-1}$

Symbol	Description	Unit, value of constant
T	temperature	K
ΔT	temperature amplitude of second sound	K
ΔT_{2s}	measured temperature amplitude with TES	K
t	time	s
t_0	start time of activation of the heater	s
t_{2s}	detection time of second sound with sensor	s
u_1	velocity of first sound	ms^{-1}
u_2	velocity of second sound	ms^{-1}
u_A	measurement uncertainty type A	
u_B	measurement uncertainty type B	
u_c	combined measurement uncertainty	
V	voltage	V
V_{av}	average measured signal	V
V_n	noise level	V
V_{2s}	measured signal due to second sound	V
v_n	velocity of normal fluid component	ms^{-1}
v_s	velocity of superfluid component	ms^{-1}

Greek letters

Symbol	Description	Unit, value of constant
α	angle heater-sensor	$^\circ$
β	angle measured outside cryostat	$^\circ$
Γ_2	amplitude parameter of second sound	$\text{m}^3\text{W}^{-1}\text{s}^{-1}$
γ	ratio normal to superfluid component	
Δ	gap energy of Cooper pairs	W
ϵ_0	electric constant	$8.854 \times 10^{-12} \text{Fm}^{-1}$
ζ	nominal sensitivity of a sensor	
η	dynamic viscosity	Pa s
ξ	measured peak intensity of second sound	
ρ_b	density He-II	kgm^{-3}
ρ_e	electric charge density	Cm^{-3}
ρ_n	density of normal fluid component	kgm^{-3}
ρ_s	density of superfluid component	kgm^{-3}
τ	time of flight	s
ψ	signal-to-noise-ratio	
ω	frequency	Hz

Abbreviations

Symbol	Description
CAD	Computer-aided design
CERN	European Organisation for Nuclear Research
CMi	Center of Micronanotechnology
EDS	Energy-dispersive X-ray spectroscopy
LHC	Large Hadron Collider
OST	Oscillating superleak transducer(s)
RF	Radio frequency
SEM	Surface electron microscopy
SMD	Surface-mounted-device
SNR	Signal-to-noise-ratio
SRF	Superconducting radio frequency
TES	Transition edge sensor(s)
TOF	Time of flight

1. Introduction

1.1 CERN

The European Organisation for Nuclear Research (french: Conseil Européen pour la Recherche Nucléaire, CERN) was founded on the 29th of September 1954 by twelve European states [1]. Since 1953, residence and Laboratory of CERN are located in Geneva. CERNs main purpose is the study of fundamental particle physics by operating research facilities like particle detectors and accelerators, as well as by international collaboration in the high energy field. Nowadays, CERN has 22 member states and is one of the most important pillars of modern science. The main components of CERN's outstanding particle accelerator and detector complex are shown in Fig. 1.1. A large interacting network of cryogenic cooling, vacuum systems and electric engineering ensures the successful operation of this complex.

The currently largest particle accelerator at CERN is the 27 km long Large Hadron Collider (LHC). In the circular LHC, beams of protons or lead ions are accelerated by sixteen 400 MHz superconducting radio frequency cavities to almost the speed of light. More than 1000 superconducting magnets ensure the correct trajectory of the particles. To be able to operate cavities and magnets properly, the devices are cooled down by liquid helium to a temperature of 4.5 K and 1.9 K, respectively [2]. When the particles have the desired energy, the particle beams are made to collide in one of the different detector facilities. During the high energy collision, subatomic particles are created which are then tracked and measured by the detector system. Studying these subatomic particles leads to a better understanding of atomic theory and the elementary structure of the universe.

A recent achievement of CERN's particle science is the experimental evidence of the Higgs Boson in 2015 [3]. One of the currently most important projects at CERN is the upgrade of the LHC, which allows a higher rate of collisions [4]. As part of this so-called High Luminosity Large Hadron Collider (HL-LHC), the magnet and cavity infrastructure of the LHC is being enhanced.

1.2 Quench localisation in SRF cavity testing

In the field of particle accelerators, superconducting radio frequency (SRF) cavities have become one of the leading technologies. Future projects ask for a continuous improvement of the cavities' performance. The overall efficiency of the cavity can be enhanced by maximizing the accelerating field while reducing the energy losses by heat dissipation. Hence, superconducting materials such as niobium have been used to increase the performance by several orders of magnitude.

One of the limitations in the operation of an SRF cavity is the *quench*, i.e. the loss of its superconducting state due to overheating. In some cases, there may be a spot in the cavity

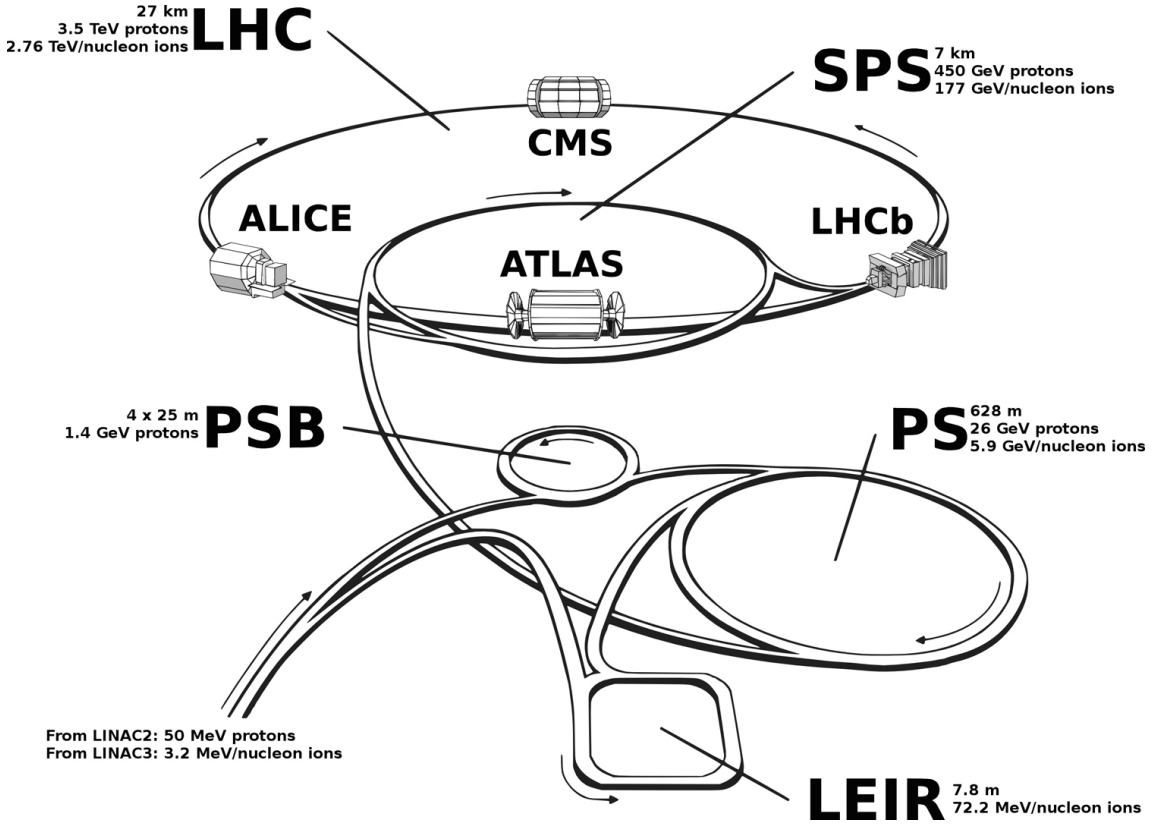


Figure 1.1: The main parts of CERN's particle accelerator and detector complex from [5]. The particles are passed from one to the next bigger accelerator (LEIR, PS, PSB, SPS and LHC) in order to obtain a velocity close to the speed of light. The collisions of the beams take place in one of the four big particle detectors ATLAS, ALICE, CMS and LHCb. It has to be mentioned that the actual CERN complex consists of much more accelerating facilities than displayed.

which dissipates more heat than the surroundings due to a surface defect, producing a local temperature rise. If the cooling is not sufficient or the localised power deposition is too high, a thermal runaway can happen that leads the hole cavity to quench. Small material imperfections on or below the inner surface of the cavity can lead to a local concentration of the magnetic field. As a result, the resistance of the surrounding area increases significantly causing the loss of superconductivity at this spot much earlier than theoretically expected.

To avoid this, high fabrication standards and a thorough testing period are necessary to ensure the maximum performance level of the cavity. A crucial step during the validation of an SRF cavity is to locate the starting point of the quench in order to understand it and correct it.

Since many SRF cavities are operated in a superfluid helium bath at about 1.8 K to achieve higher performance, the cavities are tested in such a superfluid helium bath, too. A quench can cause a thermal wave along the superfluid helium, the so-called *second sound*. Detecting this wave and its time of flight with several detectors, the time-shift information obtained can be used to track the localisation of the quench precisely by trilateration.

Nowadays, the most commonly used devices for second sound detection in SRF cavity tests are oscillating super-leak transducers (OST). However, the application of OST as a quench

localisation system has some disadvantages, regarding the accuracy of the detection or the fact that OST are not able to measure temperature fluctuations directly.

Another technology for second sound detection are the so-called transition edge sensors (TES). TES are thin-film thermometers with fast response time which consist mainly of a metallic bilayer of a normal conducting metal and a superconductor. Small temperature changes can be detected with the TES, since the resistance of the sensor increases steeply within the superconducting-to-normal conducting transition. The range of the transition is around 200 mK, but can be easily shifted along the temperature range by biasing the sensor with a certain value of electrical current. Recent studies at CERN [6] have shown that TES are promising candidates for replacing OST as a quench localisation system.

1.3 The contribution of this Master thesis

In this work TES of different compositions are produced by a microfabrication process based on a combination of photolithography technique and metal evaporation. On the one hand, the sensors are subject to metallurgical studies through SEM techniques. On the other hand, they are tested in a saturated He-II bath to measure their characteristic behaviour as a superconductor. Thus, microscopic properties of the sensors can be correlated to their performance as superconductors.

After the validation, TES are assessed as second sound detectors by producing short heat pulses with planar heaters in a He-II bath imitating a quenching hotspot. Furthermore, a study is started to understand how second sound propagates in different directions with respect to a planar finite heater. This is done by investigating the TES response to second sound for different angles while changing parameters like distance and heater size.

Finally, the experiments are repeated with OST under the same conditions to compare the performance of the two types of second sound detectors.

2. Scientific background

2.1 Superconductivity

In 1911, when investigating the electrical resistivity of pure mercury at low temperatures, H. K. Onnes observed the vanishing of the electrical resistance at 4.2 K (see Fig. 2.1). Cooled down with liquid helium, the mercury went below its critical temperature T_c and transitted from the normal to the so-called supercondcuting state. In this state, electric current can flow without any measurable resistance for direct current [7].

In metals, the electric current is conducted by a large quantity of free electrons. When applying voltage, the electrons get accelerated towards the point with higher electric potential. Electrical resistivity results from the interaction between these electrons and the atom lattice. Due to lattice vibrations (phonons) or defects, the electrons get scattered and lose some of their energy. The equilibrium value of current is reached when these interactions counterbalance the accelerating effect of the electric field [9].

Following that, in a superconductor, the losses of the electrons have to be zero. This phenomenom was later theoretically explained by J. Bardeen, L. N. Cooper and J. R. Schrieffler. They proposed that the electrons in a superconductor condense in so-called Cooper pairs, each consisting of two electrons with opposite spin and momentum [10]. As particles with half-integer spin, electrons normally follow the Fermi-Dirac statistics. Therefore they obey the Pauli exclusion principle, which says that two fermions can never occupy the same quantum state [11]. Cooper pairs, however, have integer spin and hence obey the Bose-Einstein statistics. As quasi bosons, all Cooper pairs can be in the ground state and suppress the energy losses to the lattice by electron-electron interaction. The proportion of the electrons in normal state or in Cooper pairs depends mainly on the temperature [12].

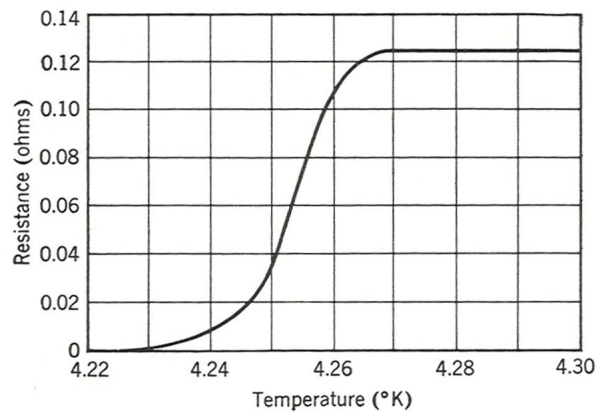


Figure 2.1: Resistance of mercury as a function of temperature showing the sharp transition from normal to superconducting state [8].

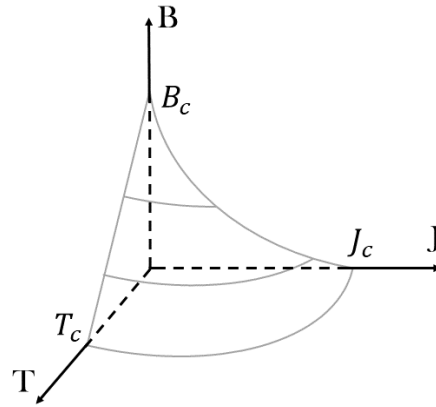


Figure 2.2: Schematic diagram of the interdependence of the three critical parameters for a superconductor.

Since the bonding energy of the Cooper pairs is finite, there are three conditions which have to be fulfilled simultaneously in the material to achieve superconductivity: temperature, current density and magnetic field. These parameters have to be below a certain limit, otherwise the Cooper pairs break and the solid transits from superconducting to normal conducting state. This transition is called “quench”. In Fig. 2.2 the correlation between the three limiting parameters is shown. The critical temperature T_c , the critical current density J_c and the critical magnetic flux density B_c are the highest values possible where the solid is still superconducting. They can be observed in the case that the other two parameters are exactly zero. If any of the remaining parameters are non-zero, the superconducting state is lost for a lower value of the parameter of interest [7].

2.2 Superconducting Radio Frequency Cavities

Superconducting radio frequency (SRF) cavities have been used for many years and are one of the key devices in particle accelerators. The most common use of them is the acceleration of charged particles by interaction with a high frequency oscillating electromagnetic field. Other purposes are the deceleration or bunching of the particle beam. Images of two cavities can be seen in Fig. 2.3.

A classical radio frequency (RF) cavity consists of a metallic conductor which defines a specially shaped hollow body. The inside of the cavity is under ultra-high vacuum to minimise the kinetic losses of the particles crossing the cavity due to interaction with other molecules. The shape and dimensions of the cavity determines the *eigenmodes*, i.e. distribution of the electromagnetic field, that can oscillate within the cavity. These are chosen depending on the purpose of the cavity and the type and velocity of the particles in the accelerator. The electromagnetic field is created and excited by coupling an antenna with an RF source. When the frequency of the RF power fed by the antenna is the same as the eigenfrequency of some modes of the cavity, the electromagnetic waves resonate within the bandwidth of around 0.1 to 1.0 Hz as typical values building up high amplitudes. Since the resonating magnetic field is continuously changing its direction, the resulting electric field, which is perpendicular to the magnetic field, does the same. Depending on the

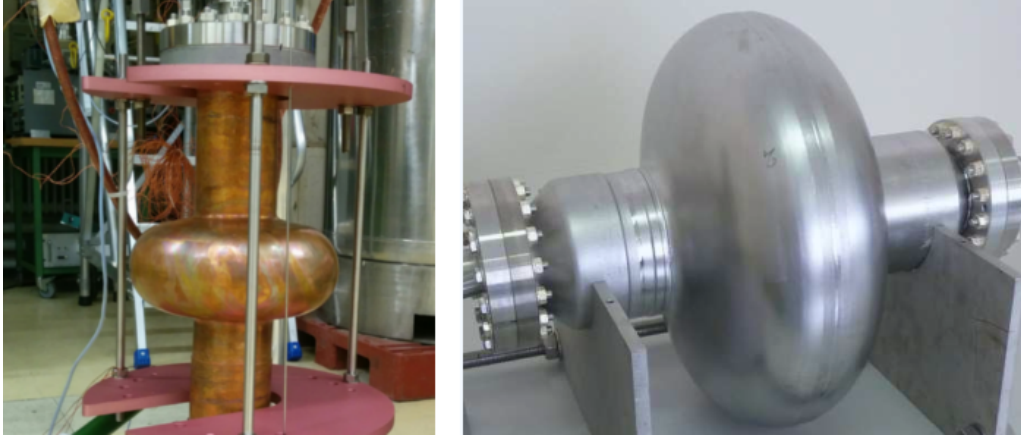


Figure 2.3: Left: An elliptical one-cell cavity made out of copper. The inner wall of the cavity is coated with a superconducting Nb_3Sn film. Right: An elliptical one-cell cavity made out of bulk Nb [13].

instantaneous direction of the electric field, the particles crossing the cavity get accelerated or decelerated. Therefore, it is important to synchronise the resonance of the cavity precisely to the instant of passage of the particles.

The electromagnetic waves inside a cavity can be described by the four Maxwell equations, here quoted in differential form [14]:

$$\begin{aligned}
 \nabla \times H &= J + \frac{\partial D}{\partial t} \\
 \nabla \times E &= -\frac{\partial B}{\partial t} \\
 \nabla \cdot D &= \frac{\rho_e}{\epsilon_0} \\
 \nabla \cdot B &= 0
 \end{aligned} \tag{2.1}$$

where H represents the magnetic H-field, J the electrical current density, B the magnetic flux density, D the electric displacement field, E the electric field, t the time, ρ_e the electric charge density and ϵ_0 the electric constant [14].

In order to create standing electromagnetic waves inside a cavity, the waves have to be reflected on the inner surface of the cavity. A perfect conductor would reflect the waves with zero loss, giving the boundary conditions that are used for the calculation of the eigenmodes: $E_{\parallel} = 0$ and $B_{\perp} = 0$. The simulated electric and magnetic field inside an elliptical cavity is shown in Fig. 2.4.

However, one finds that the eigenmodes are associated to time varying current sheets on the internal surface of the cavity. Real materials, even superconductors, require electric field parallel to the surface to withstand oscillating current density. This coupled with the impossibility of having infinite current density results in a penetration of the wave in the material. The interaction of the wall material's electrons with the crystalline lattice converts electromagnetic energy into heat. To minimize these losses, a highly conducting metal should be used for cavities. Classic RF cavities are mainly made out of copper. To reduce the losses further, a superconductor can be chosen, resulting in a SRF cavity [15].

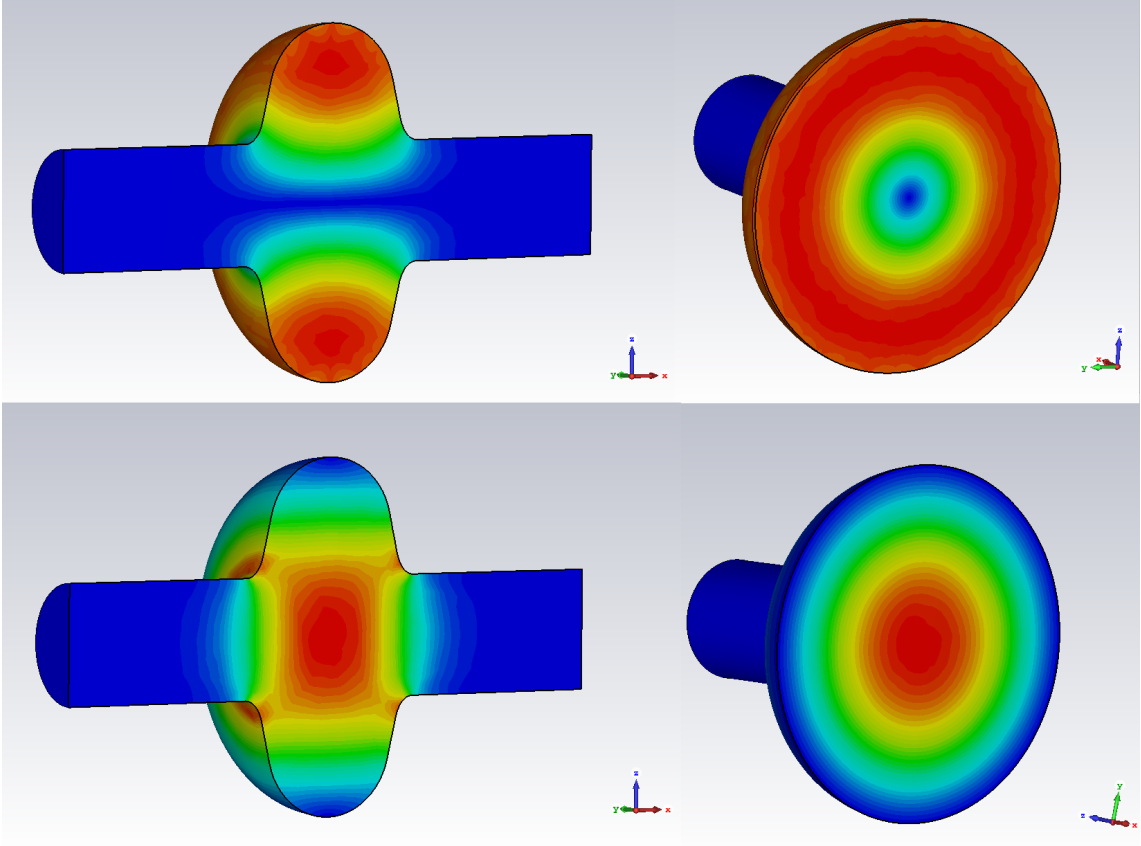


Figure 2.4: Upper row: Simulated magnetic field inside an elliptical 1.3 GHz one-cell cavity, where red indicates the highest value and blue the lowest. Lower row: Simulated electric field inside an elliptical 1.3 GHz one-cell cavity, where red indicates the highest value and blue the lowest. For each field, two cross sections are displayed. The particles pass the cavity along the rotation axis shown in the left images. Courtesy of M. Arzeo.

Most SRF cavities are made of bulk niobium (Nb), Nb coated with Nb₃Sn or bulk copper which is coated with a niobium film. Although the cavity is superconducting, there is still energy dissipation for $T > 0$ K when alternate field is applied. The problem of the response of a superconducting material to electromagnetic RF waves has been deeply studied during the 20th century. Taking into account the presence of normal electrons and Cooper pairs, the parameter known as *RF surface resistance* can be determined to quantify the heating losses in the superconductor. From [16], the RF surface resistant R_S can be expressed by

$$R_S = R_{BCS}(T) + R_0 = A_s \omega^2 e^{\frac{-\Delta}{k_B T}} + R_0 \quad \text{for } T < \frac{T_c}{2}. \quad (2.2)$$

The surface resistant consists of two parts. The first term describes the losses which depend on the frequency of the electromagnetic field ω , the superconducting energy gap Δ above which the Cooper pairs break and material parameters, summed up in the factor A_s . R_0 represents the temperature independent residual resistance, which is caused by imperfections, for example impurities or crystalline defects. It is normally in the order of 10 nΩ for Nb-cavities [17].

The resulting heat flux from the vacuum into the wall due to the interaction of the electro-



Figure 2.5: Picture of a surface defect detected on an SRF cavity [18].

magnetic waves with the material can be locally calculated as:

$$\dot{q}_S = \frac{1}{2} R_S |H|^2 , \quad (2.3)$$

where $|H|$ is the local magnetic field peak intensity on the surface of the wall.

The limiting factors of an SRF cavities are the critical parameters of its superconducting material. If the critical values are reached, the materials start to quench at a certain spot. A cavity without defects would always quench at a certain field because of thermal feedback and critical magnetic field (see Section 2.1). Increase of field means increase of power on the wall causing an increase of temperature, until the critical condition is reached at the point with highest magnetic field.

It could also happen that a localised surface defect enhanced the magnetic field (see Eq. (2.3)) or R_S were increased because of local bad cooling. If the level of field in the cavity is increased, the hotspot becomes unstable, and the quenching area spreads. This leads to a thermal runaway until the complete cavity is normal conducting.

In practice, the quench often happens earlier than theoretically expected. The reason for that is generally the presence of localized defects, as the one shown in Fig. 2.5. Due to the local magnetic field enhancement, the material at this spot transits already at lower accelerating electric field from superconducting to normal conducting state [18]. That is why it is essential to detect and localise these imperfections and correct them.

To achieve superconducting state and extract the heat deposited on the internal walls of the cavity, SRF cavities have to be operated in a *cryomodule* [16]. There, they are cooled below their critical temperature by immersion in a liquid helium bath [19].

2.3 Helium

Helium (He) is the lightest noble gas and the second-lightest element in the periodic table. It exists in the form of two stable isotopes, ^3He and ^4He . Both play important roles in cryogenics. As shown in Fig. 2.6, ^3He and ^4He are the only two elements which have boiling points below 10 K. Their boiling temperatures at atmospheric pressure are 3.191 K and

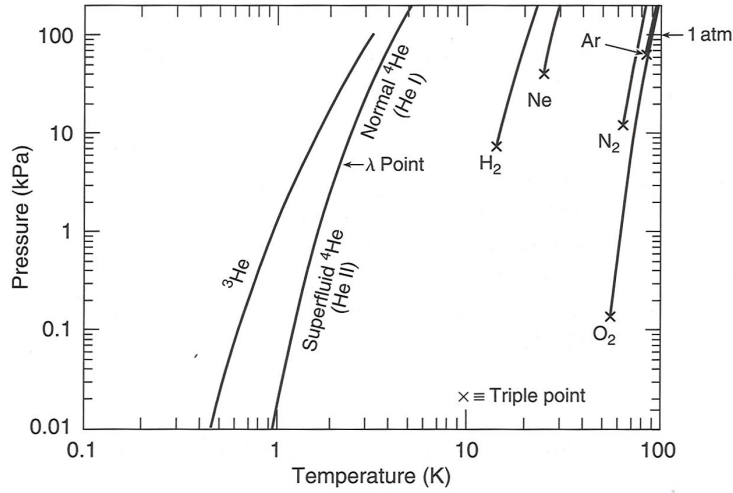


Figure 2.6: Vapour pressure curves for several cryogenic fluids obtained by vacuum pumping or pressurising. The lowest temperatures can be reached with ^4He and ^3He [20].

4.230 K, respectively [20]. While ^4He is a common liquid coolant for general cryogenic applications, the much rarer ^3He is usually only used for specific low-temperature measurements below 1 K, for example in dilution refrigerators [21]. In this work, the expression “helium” always refers to ^4He . Helium has several remarkable properties, as it can be seen from its phase diagram (Fig. 2.7). One is the fact that helium is the only element which can not be solidified at atmospheric pressure [22, 23]. This can be explained by the large zero-point energy of helium. In a simple approach, the zero-point energy E_0 of a molecule can be expressed as

$$E_0 = \frac{h^2}{8m(\frac{\bar{V}}{N_A})^{2/3}}. \quad (2.4)$$

In this equation h is the Plank constant, m the mass of the molecule, $\bar{V}$ the averaged volume and N_A the Avogadro constant. Due to helium’s low mass, its potential binding energy is lower than the zero-point energy. Thus, external pressure is necessary to solidify helium. Hence, it has no triple point [24].

Helium has two different liquid phases helium I (He-I) and helium II (He-II), seperated by the so-called λ -line. It has its name after the characteristic shape of the plot of specific heat C as a function of temperature near the transition, which looks like the Greek letter λ (see Appendix A.1) [25].

He-I follows the regularities of a classical fluid. However, due to its weak intermolecular potential, helium I has the lowest critical point of all elements. Most cryogenic applications which use He-I operate at saturated vapour pressure, where liquid and vapour phase are at equilibrium. To reach lower temperatures, the pressure of the vapour phase above the liquid is decreased by vacuum pumping. This operation can be pictured in Fig. 2.7 by following the vapour pressure curve to lower temperatures[25, 26]. If the pressure of the saturated helium is reduced below 5036 Pa, which means $T < 2.178 \text{ K} = T_\lambda$, the liquid transits from He-I to He-II [23].

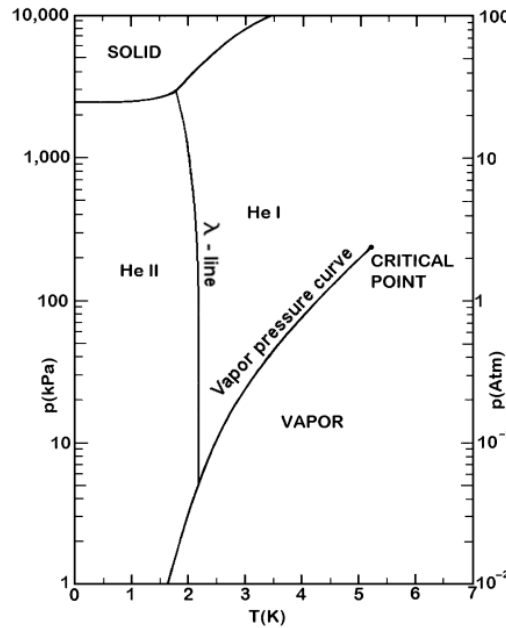


Figure 2.7: $p - T$ -phase diagram of ${}^4\text{He}$, from [25].

2.3.1 Helium-II

He-II has exceptional properties, such as a thermal conductivity which is orders of magnitude higher than that of any other fluid or the ability to flow through tiny capillaries without any friction [27, 28]. This phenomenon, called superfluidity, can not be explained with simple fluid mechanics. In 1938, a first proposal was made by F. London, who connected the λ -transition between He-I and He-II to Bose-Einstein condensation [22, 29]. He compared He-II to an ideal gas of non-interacting bosons. Below a critical temperature T_c , a macroscopic number of particles occupy the zero momentum state due to Einstein-Bose statistics. For helium this temperature is T_λ . As long as the excitation energy is below a certain threshold, the bosons in the ground state, acting as a single macroscopic quantum current, can not be excited. Thus, it is not possible to transfer energy to the fluid, resulting in a frictionless flow. This behaviour is comparable to that of Cooper pairs in superconductivity.

Although many properties of He-II can be explained with this theory, it has obviously some shortcomings. Contrary to a non-interacting Bose gas, helium shows repulsive and attractive forces between its particles, depending on the particle distance. This results in an imprecise calculation of T_λ and the specific heat, for example.

Still in 1938, based on London's theory, L. Tisza published the two-fluid model [30]. This phenomenological theory describes He-II as a mixture of two different interpenetrated components, the normal fluid of density ρ_n and a superfluid of density ρ_s . The bulk density of He-II ρ_b can then be described as the sum of the densities of the two components [24]

$$\rho_b = \rho_n + \rho_s . \quad (2.5)$$

The superfluid component, a single quantum state, has no entropy ($s_s = 0$) and zero viscosity ($\eta_s = 0$). Therefore, the normal component must carry all the entropy of the bulk

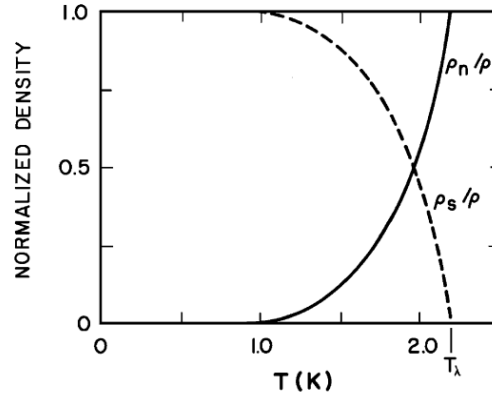


Figure 2.8: Ratio between the normalised densities of the normal and superfluid component in He-II [25].

fluid s_b :

$$s_b = \frac{\rho_n}{\rho_b} s_n, \quad (2.6)$$

where s_n is the specific entropy of the normal fluid. Thus, it also determines the viscosity of He-II, η_b :

$$\eta_b = \eta_n. \quad (2.7)$$

For $T = 0$ K all the helium molecules are in the ground state. As T increases, thermal excitations are created in the fluid, represented by an increase of the relative amount of normal fluid. At T_λ , the bulk fluid only consists of the normal fluid component, as it is shown in Fig. 2.8.

A quantitative relationship between T and $\frac{\rho_n}{\rho_b}$ is given by [25]

$$\frac{\rho_n}{\rho_b} = \left(\frac{T}{T_\lambda} \right)^{5.6} \quad \text{for } 1.1 \text{ K} \leq T \leq T_\lambda. \quad (2.8)$$

based on the assumption that the two components have their individual velocities v_s and v_n .

The hydrodynamical behaviour of He-II can be approximated by the Navier-Stokes equations for the two-fluid model [31]:

$$\rho_n \frac{\partial v_n}{\partial t} = -\frac{\rho_n}{\rho_b} \nabla p + \rho_s s_b \nabla T + \eta_b \nabla^2 v_n \quad (2.9)$$

$$\rho_s \frac{\partial v_s}{\partial t} = -\frac{\rho_s}{\rho_b} \nabla p - \rho_s s_b \nabla T, \quad (2.10)$$

where p is the pressure. As it can be seen, the two equations are connected via a hydrothermical term $\rho_s s_b \nabla T$ which depends on the temperature gradient. The momentum density of helium II j_b can be described as

$$j_b = \rho_s v_s + \rho_n v_n. \quad (2.11)$$

It can be seen that for the specific case $j_b = 0$, when there is no net mass flow, Eq. (2.11) results in

$$\rho_s v_s = -\rho_n v_n . \quad (2.12)$$

The heat flux density $\dot{q}$ is proportional to the velocity of the normal component [32]

$$\dot{q} = \rho_b s_b T v_n . \quad (2.13)$$

The fact that a temperature gradient is converted into momentum is the most remarkable property of He-II. A heat source in He-II will force the superfluid to flow towards it while the normal fluid, which carries all the thermal energy of the system, moves away from it [33]. This counterflow of the two components allows the system to transport entropy, although the bulk He-II stays static on a macroscopic level. However, the thermal conductivity values of He-II expected by this model are much higher than the ones which were actually measured [34].

Finally, it has to be said that despite giving an explanation for many occurring features of He-II, the two-fluid model is only a theoretical assumption. There are no distinguishable particles in He-II.

2.3.2 Second Sound

One of the achievements of Tisza's two-fluid model was the prediction of propagation of temperature-entropy waves with constant velocity. Later, this phenomenon was given the name of "second sound" by Lev Landau [35]. He had a different ansatz from Tisza's by explaining the temperature dependent superfluidity of He-II with excitations called "rotons" and "phonons" [36]. However, both theories resulted in the conclusion that there have to be two different components in He-II with individual velocity fields, as shown in the previous section. The first experimental detection of second sound was made by V. Peshkov in 1944 [37].

After some manipulation and neglecting the viscous term, it can be shown that Eq. (2.9) and Eq. (2.10) result in two wave equations [31]

$$\frac{\partial^2 \rho_b}{\partial t^2} = \nabla^2 p \quad (2.14)$$

$$\frac{\partial^2 s_b}{\partial t^2} = \frac{\rho_s s_b^2}{\rho_n} \nabla^2 T . \quad (2.15)$$

Thus, there have to be two velocities of "sound" in He-II [36]

$$u_1^2 \approx \left(\frac{\partial p}{\partial \rho} \right)_s \quad (2.16)$$

$$u_2 \approx \sqrt{\frac{T s_b^2 \rho_s}{C \rho_n}} . \quad (2.17)$$

The velocity u_1 given in Eq. (2.16) represents the classic first sound, often called speed of

sound. In first sound, the normal and superfluid component move in phase with each other. Pressure changes lead to fluctuations of the bulk density, as shown in Eq. (2.16). The value of first sound in He-II lies around 230 m/s and is only weakly dependent on temperature [38, 39].

The expression for second sound after Eq. (2.17) was first published by Landau [35]. This equation only applies if the heat capacities at constant pressure c_p and volume c_v are almost equal [37]

$$c_p \approx c_v \approx C, \quad (2.18)$$

which is the case for liquid helium.

The different ways in which first and second sound propagate are schematically compared in Fig. 2.9. The ratio between the normal and the superfluid component is represented by the proportions of the letters n and s . The bulk density of the fluid is represented as the total number of letters in a column. In the picture, the waves are harmonic and travel in horizontal direction. As already mentioned, first sound propagates thanks to changes in the bulk density. In second sound waves, the bulk density is constant to first order, while the relative portion of the two fluids varies in the direction of propagation due to the action of a thermal gradient. This results directly from the counterflow model explained in the previous subsection. The normal fluid component, which carries all the thermal energy, is accelerated against the thermal gradient resulting in a propagating thermal wave with constant speed [37]. As suggested by on Eq. (2.17), the second sound velocity strongly depends on the temperature. For $T = T_\lambda$ the speed of second sound becomes zero, because without superfluid there is no ratio difference of the two components which could propagate [35].

Up to date, second sound has been extensively studied and the values of the velocity over a wide temperature range are well known [40, 41, 42, 43]. Most of these authors measured the speed of second sound with the so-called resonance method. In this measurement setup, a standing second sound wave is generated by periodical heating in a small tube. Detecting the peaks and nodes of this pattern, the velocity can be calculated precisely. In Fig. 2.10 the propagation speed of second sound for $0.5 \text{ K} < T \leq T_\lambda$ is shown [43]. Between 1.0 K and 2.0 K the velocity of second sound is in the range of 16 m/s to 20 m/s, whereas it steeply increases for lower temperatures.

Amplitude dependence of second sound velocity

An effect that has to be taken into account about the second sound velocity is its amplitude dependence, first published by Temperly [44] and Osborne [45]. This concept describes the correlation between the second sound velocity and the density of the heat flux. Over the years this theory has been developped [46] and finally experimentally proved by Dessler and Fairbank [47]. According to Eq. (2.13), the velocity of the normal fluid is proportional to the heat flux $\dot{q}$ which explains the dependence especially for higher heat pulses. A calculation of this effect based on empirical data is given by Dessler and Fairbank [47]:

$$u_2 = u_{2,0} + \Gamma_2 \dot{q}, \quad (2.19)$$

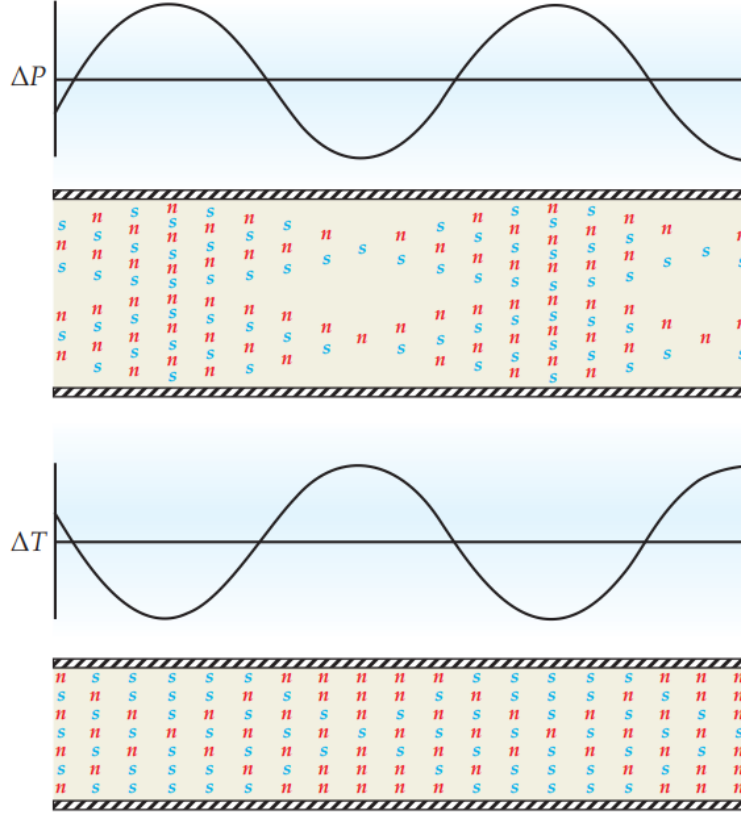


Figure 2.9: Schematic comparison of first (top) and second sound waves (bottom) in He-II, where the normal fluid and superfluid components are depicted as n and s , respectively [37].

where $u_{2,0}$ is the speed of second sound for an infinitely small heat pulse and Γ_2 the amplitude dependence parameter. It is only dependent on the temperature and helium properties. In Fig. 2.11 the plot of Γ_2 over T measured by Dessler and Fairbank is shown.

Finally, it is important to mention that these experiments were done in a small tube with constant cross section. As a consequence the heat flux density is constant over the complete measured distance. For this reason it is questionable if the results of this simplified case also apply for non-confined geometries in three dimensions [13, 48].

3D propagation of second sound

In the case of three dimensional propagation of second sound, the behaviour and shape of the wave complexify. Iznankin and Mezhev-Deglin observed the propagation of spherical shock waves [49]. A mathematical explanation for this observation was made by Atkins for a linear and non-linear case, depending on the heat flux [50]. Based on the equations of motion (Eq. (2.9) and (Eq. (2.10)) and correlation between heat flux and velocity of the normal component (Eq. (2.13)), he calculated the temperature amplitude ΔT of the outgoing spherical wave

$$\Delta T = r^{-1} g' \left(t - \frac{u_{2,0}}{r} \right), \quad (2.20)$$

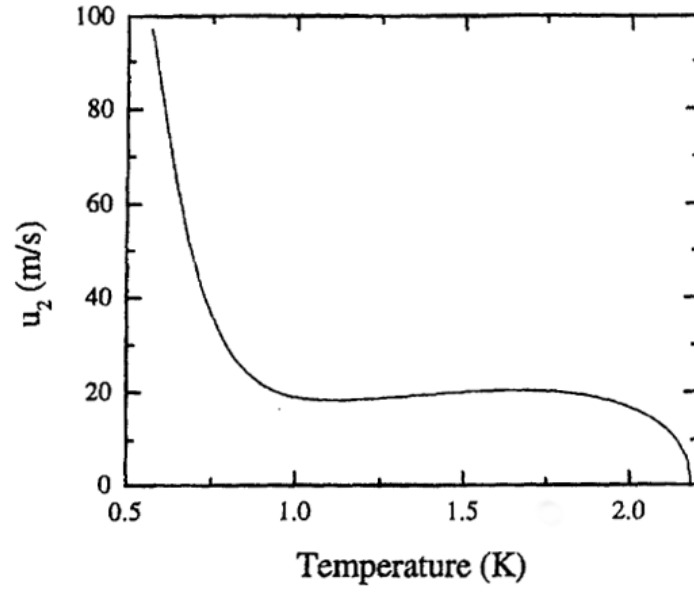


Figure 2.10: Propagation speed of second sound u_2 over temperature at saturated vapour pressure [43].

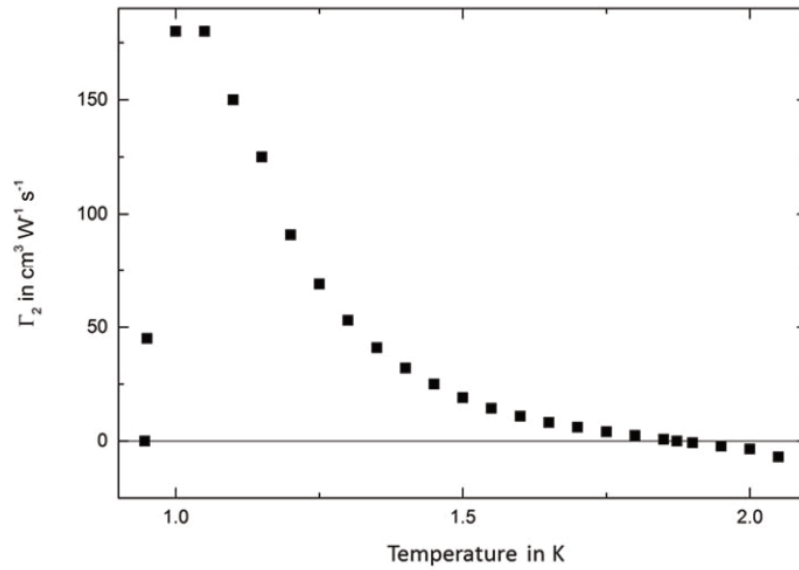


Figure 2.11: Measured amplitude dependency parameter Γ_2 over T by Dessler and Fairbank, replotted [48].

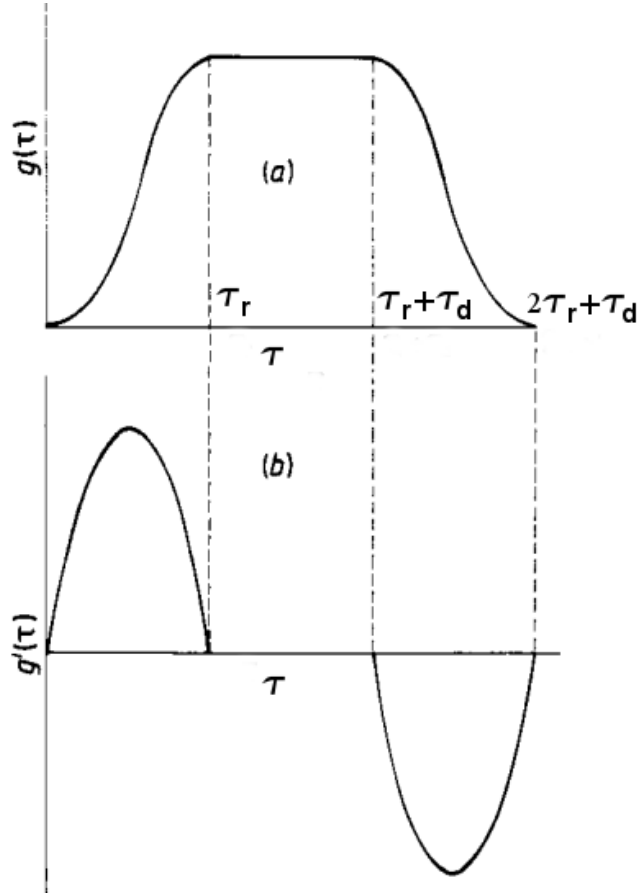


Figure 2.12: Construction of the thermal profile of a second sound wave (b) relative to the bath temperature based on the heat flux (a) after Atkin [50].

where g is a function which can be determined from the boundary conditions, represented by the history of heat flux on the surface of the heater, and r the distance from the center of the spherical heater. These equations indicate that for the linear case the amplitude of the temperature wave propagates with constant speed $u_{2,0}$ and decays with r^{-1} . Under the assumption of an infinitely small, spherical heat source follows

$$g(t) = \frac{Q(t)}{4\pi\rho_b T s_b^2 \gamma} \quad (2.21)$$

where $Q(t)$ represents the total rate of heat supply, T the surrounding bath temperature and γ the ratio of superfluid to normal fluid densities. Atkins showed that the shape of the propagating wave can be directly deviated from the time-dependent profile of the heat pulse. This is shown in Fig. 2.12 for a simple symmetric pulse of a duration τ_d with a certain rise and decaying time τ_r . According to this calculation, the heat pulse produces an outgoing wave with a positive temperature disturbance in front followed by a negative one. This phenomenon has been experimentally proved in several publications, for example [51].

Thanks to the big computing power of computers nowadays, it is possible to simulate the propagation of second sound waves in 3D. This was done by Zhang and Mukamari, who calculated the second sound propagation in He-II caused by a finite plane heater [52]. An excerpt of their results at 1.6 K is shown in Fig. 2.13. The figure displays 2D

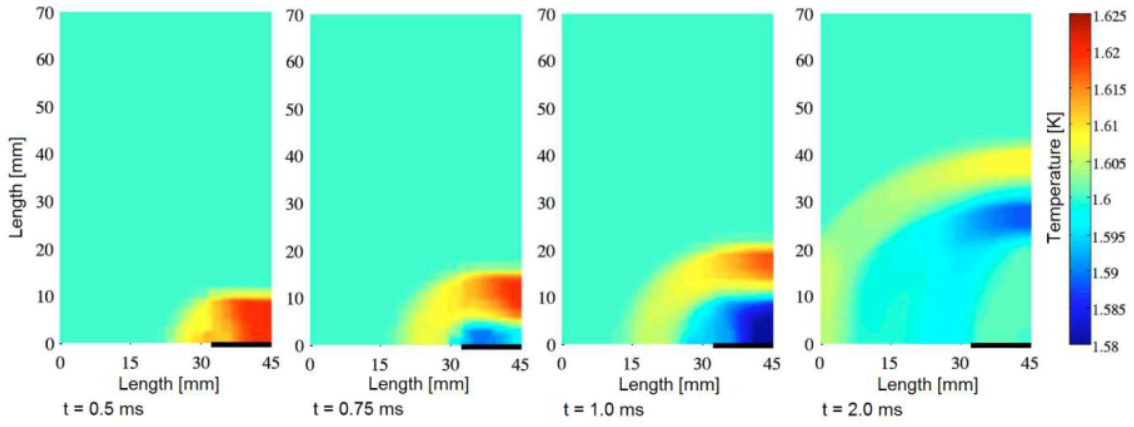


Figure 2.13: Propagation of a 3D second sound wave emitted by a $25 \text{ mm}^2 \times 25 \text{ mm}^2$ planar heater with a heat flux of 100 kW/m^2 at a bath temperature of 1.6 K . From [52], rearranged by [13].

slices of the temperature field at different times. The first slice is taken at the end of the heating pulse and the created temperature rise in front of the heater is clearly visible. With increasing time the temperature of the wave, which propagates almost spherically, decays. The temperature amplitude in the region perpendicular to the heater plane is significantly higher. Also the cooling front, which evolves at the edge of the heater, is visible. This is in accordance with Atkin theory and can be deduced from the simple theoretical model for the perfect spherical heater illustrated in Fig. 2.12.

2.4 Quench Thermal Mapping

2.4.1 Contact thermometry

As it has been pointed out in Section 2.2, it is indispensable to extensively test and investigate a SRF cavity and its performance before mounting it in the final application. An important part of this investigation is the thermal mapping. When the cavity is driven to the limits of its performance, sooner or later a quench will occur. To understand the cause of the initiated quench, the originating point of the quench has to be localised.

A way to do this is measuring the temperature on the surface of the cavity. A large amount of thermometers are uniformly distributed over the cavity and placed on its external surface. By this the temperature field of the whole cavity can be monitored. When a quench occurs, the thermometers close to the hotspot will measure the highest temperature increase indicating the origin of the quench. Moreover, the spread of the quench over the cavity can be seen [53, 54].

The fact that the tests are done in liquid helium at very low temperatures limits the selection of possible thermometers for thermal mapping. A common device used is the Allen-Bradley carbon resistor [55]. It is able to operate at low temperatures with high sensitivity, can easily be glued on the surface and has a sufficiently fast time response [56]. Fig. 2.14 shows the results of a thermal mapping of a cavity conducted with Allen-Bradley

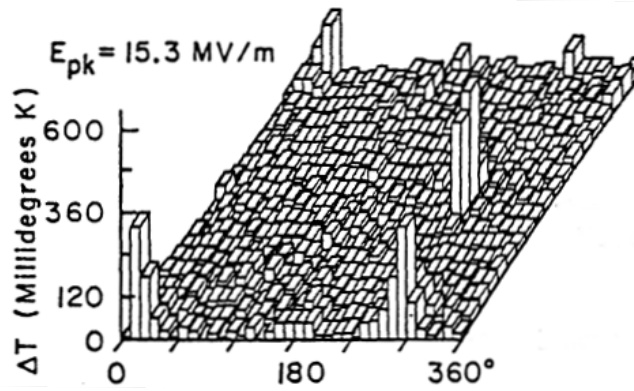


Figure 2.14: Radial temperature map of a 1500 MHz SRF cavity with an electric field of 15.3 MV/m at 1.5 K, from [57].

carbon resistors. The large peaks clearly identify the spots where field emission heating occurs.

Although thermal mapping done by contact thermometry yields detailed information about the cavity, there is obviously one big disadvantage: Each cavity has to be prepared individually and sometimes up to 200 thermometers have to be glued on the cavity wall manually. In the last decades, several techniques and devices have been invented to accelerate and automatise this method [58]. But the continuous development of SRF cavities in size and shape plus the increasing demand [59] limits the effect of these improvements considerably.

2.4.2 Oscillating Superleak Transducers

In order to bypass the contact thermal mapping, devices with the ability to locate quenches at a distance have been created. This can be done by tracking second sound waves emitted by the normal conducting hot spot at the moment of its onset with an appropriate detector. The most widespread tool for this method is the Oscillating Superleak Transducers (OST). An OST is a capacitor consisting of a metallic chassis with a semi-permeable metal coated membrane [60] (see Fig. 2.15).

The working principle of an OST is similar to that of a microphone. The superleaking membrane is covered with microscopic holes, through which only the superfluid component of the helium can flow due to its non-existing viscosity. A second sound wave propagates by counterflow of the normal and superfluid component. When the heat front of the wave, which has a higher proportion of normal fluid, arrives at the OST, an imbalance of flow between the two components forces the membrane to deform. While the superfluid diffuses out of the OST through the membrane attracted by the second sound thermal gradient, the normal fluid cannot penetrate the superleaks, which originates a pressure difference. The resulting movement of the membrane with respect to the electrode of the OST changes its capacitance [61]. Normally, a downstream electronic setup then filters the noise and amplifies the signal [13]. The principle of second sound detection with OST is also shown in Fig. 2.15.

In the case of an occurring quench, the start of the event can be noticed by a sudden drop of the RF power. If several OST are placed around a cavity, they will detect the second

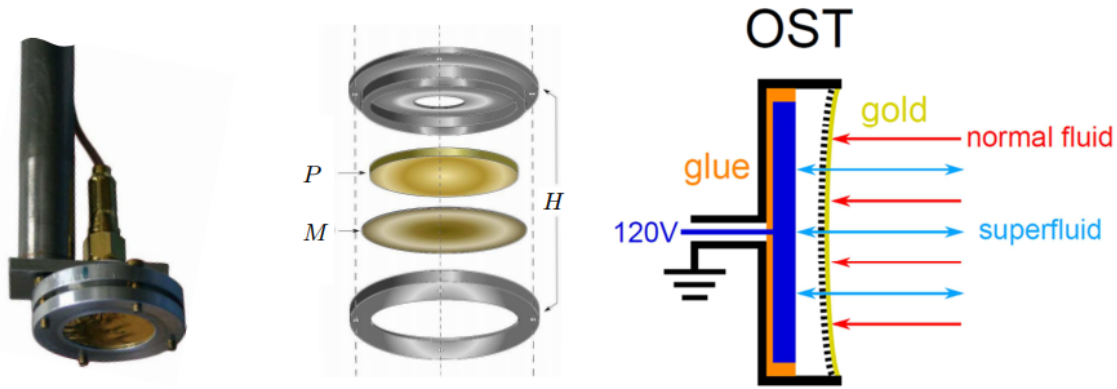


Figure 2.15: Mounted OST (left) and drawn components (middle) with H = housing, M = porous membrane and P = second electrode [62]. Right: Working principle of an OST [63].

sound at varying time, depending on their distance to the quench spot. By knowing the speed of the propagating wave at the bath temperature, the distance to the quench can be calculated with the measured time of flight (TOF). The TOF of the wave is the time difference between the drop of the RF power of the cavity and the detection of second sound by the OST. By knowing the position of at least three OST with respect to the cavity and the distance to the second sound emitter, the hotspot can be located within a precision of a few centimeter by simple algorithm like trilateration [64].

Despite the fact that OST are very successful in second sound detection, there lack a direct temperature measurement. Although the relative velocity of the two fluid components is related to the temperature over the heat flux (see Eq. (2.13)), the exact relation can only be determined by numerical simulation and also depends on the detecting angle. Furthermore, the minimal size of the membrane (1-2 centimetres) limits the spatial resolution of the detection. Observed oscillating features due to eigenmodes of the membrane complicate the measurement as well [62].

2.4.3 Transition Edge Sensors

Transition Edge Sensors (TES) are superconducting thin films which take advantage of their superconducting to normalconducting transition to measure energy deposition. The extremely sharp transition (see Fig. 2.16) from zero to normal resistance offers the possibility to use TES as highly sensitive thermometers in a narrow temperature range. This and their small size make TES superior to conventional semiconductors in terms of time response, heat capacity and resolution when it comes to the detection of very small energy input [65]. Common applications of TES are especially found in the field of astro- and particle physics, where they function as calorimeters for X-ray and particle detection [66, 67].

As it can be seen in Fig. 2.16, this type of TES has a very steep transition within a small temperature range of approximately 2 mK. It has been observed that the critical temperature T_c of a superconducting thin film can be much lower than that of its bulk material [69]. Since T_c of the superconductor depends also on the current density (see Section 2.1), the operating temperature range of the TES can be shifted towards higher

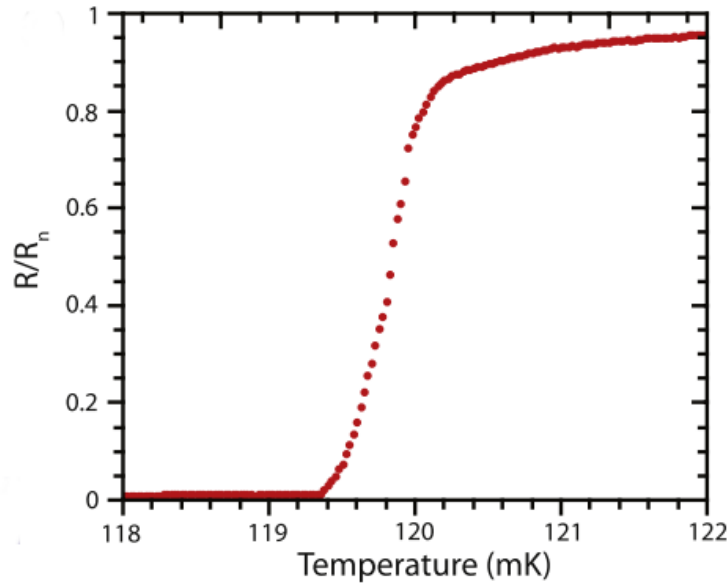


Figure 2.16: A schematic measurement of the transition of a low-temperature TES fed with a small AC current. The normalised resistance is plotted vs. the operating temperature [68].

temperatures by decreasing the bias current. The same rule applies for a magnetic field. However, the width of the temperature range of the transition is only weakly affected by this [65].

To overcome this problem, TES with a bimetallic thin film of a normal conducting and a superconducting material can be used. These sensors take advantage of the so-called proximity effect. According to this effect, Cooper pairs are able to move from a superconducting to a normal conducting film when they are directly in contact with each other. Thus, the normal conductor will become weakly superconducting while the critical parameters of the superconductor drop. As a result the overall critical temperature of the thin film decreases and the transition becomes smoother. Therefore the operational temperature interval can be easily increased to values between 200 mK to 400 mK, but simultaneously the TES loses part of its sensitivity due to the decreasing slope of the transition [70].

Transition Edge Sensors for applications in He-II

With this technique, it is simple to produce a TES for a desired temperature range by selecting a metal with appropriate T_c . For the detection of temperature fluctuations in He-II, the critical temperature of the TES has to be below T_λ . This was firstly achieved by Atkins and Rudnick in 1970 by producing a bolometer consisting of thin film of aluminium [71]. Five years later, G. A. Laguna [72] published his work about second sound detection in which he used a bilayer of 25 nm of gold (Au) followed by 100 nm of tin (Sn) as TES. The sensors were produced on a glass substrate by general microfabrication techniques, such as photolithography, thermal evaporation and etching [73].

The critical temperature of these sensors for zero field was 2.1 K and a sensitivity of 0.6 V/K. By increasing the magnetic field, Laguna was able to shift the transition temperature until

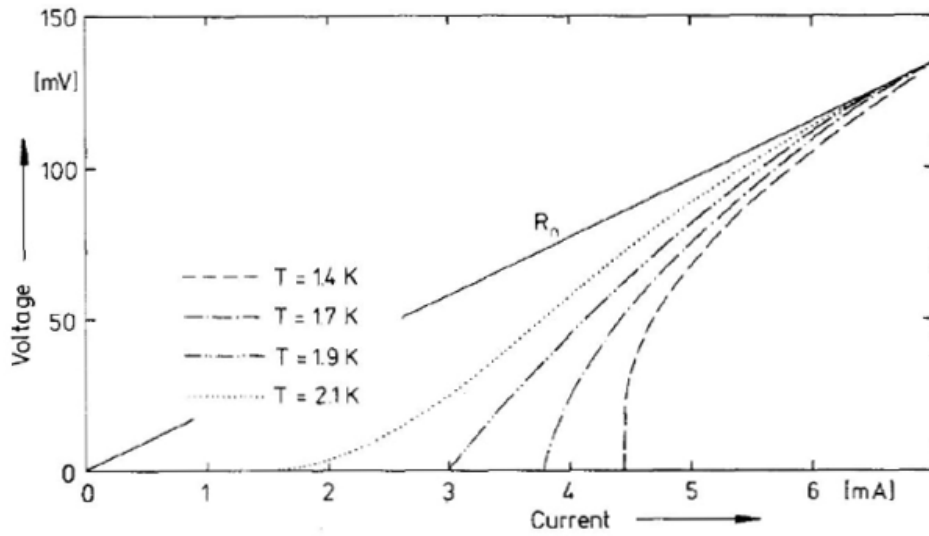


Figure 2.17: V-I curves for a 20 nm Au - 100 nm Sn thin film measured at different temperatures [75].

1.3 K, although the sensitivity dropped below 0.09 V/K. He also claimed that not only the ratio between the two metals but also the overall thickness played a crucial role in the sensor performance. TES made out of 50 nm of Sn evaporated on top of 12.5 nm of Au neither showed an acceptable transition curve for second sound detection nor reproducible results [73]. Cummings *et al.* accomplished the temperature shift of the Au-Sn sensors by only increasing the bias current and therefore avoided the use of a magnetic field. This advance made the application of TES for cryogenic experiments much more straight forward [74].

Based on all this papers, in 1982 Borner *et al.* [75] published an extensive study about Au-Sn thin films for second sound detection. They created stripes ($1300\text{ }\mu\text{m} \times 30\text{ }\mu\text{m}$) of the metallic bilayer and varied the ratio between the two metals to investigate the influence of the composition more in detail. According to their results, increasing the gold layer affected the transition much more than decreasing the thickness of the tin layer. Instead of a 4:1 proportion, they claimed the best results were achieved by films consisting of 20 nm of gold and 100 nm of tin [76]. The transition curves for this film are shown in Fig. 2.17.

Besides the influence of film composition, Borner *et al.* additionally annealed some of the samples afterwards to see how the performance changes with thermal treatment. The sensor with 20 nm Au - 100 nm Sn was therefore heated in a vacuum atmosphere at 52 °C for 15 min. The transition curves measured after the annealing are shown in Fig. 2.18. Instead of an abrupt rise in voltage at T_c , the transitions become smoother in the beginning resulting in a S-like shape. Additionally, the critical current diminished for a given temperature.

Borner *et al.* explained this by a change of the thin film's microstructure. Scanning electron microscope (SEM) images clearly showed that the untreated thin film consisted of at least two heterogeneous phases: grain-like features with a higher electron contrast and a uniform background. As it can be seen in Fig. 2.19, the fine crystalline character of the film vanished completely after annealing. There are several papers which describe the diffusion process in

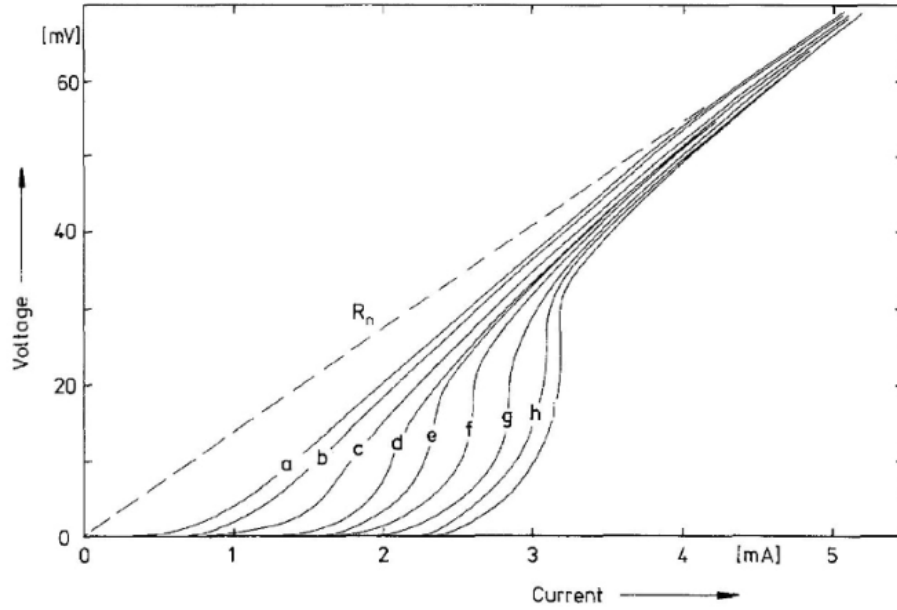


Figure 2.18: V-I curves measured for a 20 nm Au - 100 nm Sn film vacuum annealed at 52 °C for 15 min [75]. (a) 2.1 K, (b) 2.0 K, ..., (i) 1.3 K.

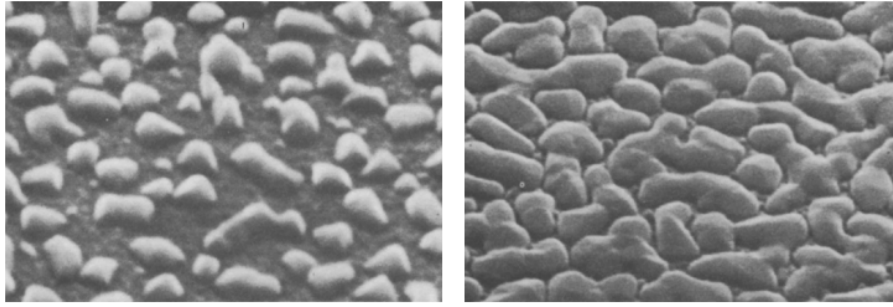


Figure 2.19: SEM images of the surface of a 20 nm Au - 100 nm Sn thin film before (left) and after vacuum annealing (right). (No scale bar was given; Magnification $\times 13,000$) [75].

gold-tin films [77, 78, 79, 80]. Buene *et al.* identified phases of the two materials according to the bulk phase diagram of Au-Sn (see Appendix. A.2) by X-ray diffraction [81]. They observed that the gold had diffused rapidly into the tin forming grains of gold-tin alloys depending on the corresponding ratio between the two deposited metals.

Based on Buene's results, Borner *et al.* suggested that the homogenous microstructure after the annealing lead to an enhancement of the proximity effect. This finally caused the less pronounced transition curve [75].

The first report about the utilisation of transition edge sensors for SRF cavity quench mapping was done in 2016 at CERN. Z. Kovács and H. Furci produced TES based on the model of Borner's work [6, 82] with different production methods and design. Although they were able to manufacture performing second sound detectors, they did not obtain the same V-I-transition curves as Borner's TES. For the same amount of gold and tin, they observed lower critical currents and less sensitivity. However, they were able to measure second sound successfully with these detectors leading to concrete projects of development

of thermal mapping systems based on TES. The work presented in this thesis is part of further research and development on these systems.

3. Materials and Methods

3.1 Fabrication of TES

The fabrication process of TES used in this work is based on the previous work of Kovács and Furci at CERN [6, 82] with some additional changes.

The production of the thin film TES was done at the Center of Micronanotechnology (CMi) of the École Polytechnique Fédérale de Lausanne (EPFL). The sensors were manufactured by means of a combination of laser *photolithography* and an *evaporation* of metallic layers followed by *lift-off*. By this method, it is possible to form geometries in micrometer scale with excellent precision. The process flow is schematically shown in Fig. 3.1. A floated borosilicate glass (Borofloat[®] 33 from SCHOTT) was used as substrate, because of its low thermal expansion and its resistance to thermal shocks [83].

Two different designs of TES have been developed at CERN in previous work. On the one hand, a hexagonal camera (HEX-TES), where 30 sensors are evenly distributed in six petals of five sensors each. This design offers the advantage of having many TES on one single device. And on the other hand, the so-called Chip-TES. For the Chip-TES, the wafer was firstly prediced in rectangles of 10 mm $\times$ 15 mm by using an automatic dicing saw (Disco DAD321). The two types of sensors are shown in Fig. 3.2.

Golden contact electrodes were included in the design to get a resistant surface for the subsequent electrical connection of the TES. This required that a first full production step were done to obtain the electrodes (made of Ti-Au), and a second one to obtain the superconducting thin films (bilayer of Au-Sn). A 20 nm titanium (Ti) layer was deposited under the gold of the electrodes to improve the adherence. A CAD drawing of the two different layers is prepared before the production. Special care is taken so that the superconducting thin film is overlapping with the electrodes to provide electric connection.

The actual sensor consists of a sensing stripe of 30 μ m width and between 6 mm and 8 mm length, depending on the design of the TES. Two leads connect the sensing stripe with the golden electrodes.

3.1.1 The photolithography process

The surface of the round borosilicate glass wafer (diameter: 100 mm, thickness: 0.5 mm) was cleaned in a high frequency oxygen plasma stripper (PVA TePla 300) for two minutes. The clean wafer was then spin-coated with a polymer bilayer of 480 nm lift-off resine (LOR 5A, MicroChem) followed by 1.1 μ m of a positive photoresist (AZ[®] 1512 HS, MicroChemicals GmbH) in an automatic coating machine (ACS 200 Gen 3). This double layer configuration is needed to help the lift-off process (see Section 3.1.3).

The CAD pattern prepared in advance with the desired shapes of the metallic elements was then written on the wafer by a Heidelberg VPG 200 photoresist laser writer. The VPG

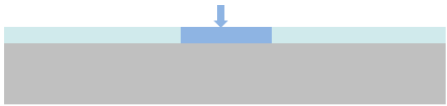
Step	Process description	Cross section after process
1	<i>Coating</i> Machine: ACS 200 Positive resist	Substrate: Borofloat 33
2	<i>LASER Writing</i> Machine: VPG 200	
3	<i>Developing</i> Machine: ACS 200	
4	<i>Metal Evaporation</i> Machine: EVA 600 Metals: Ti/Au/Sn	
5	<i>Lift-off</i>	

Figure 3.1: Process flow of the thin film production by photolithography, from [6].

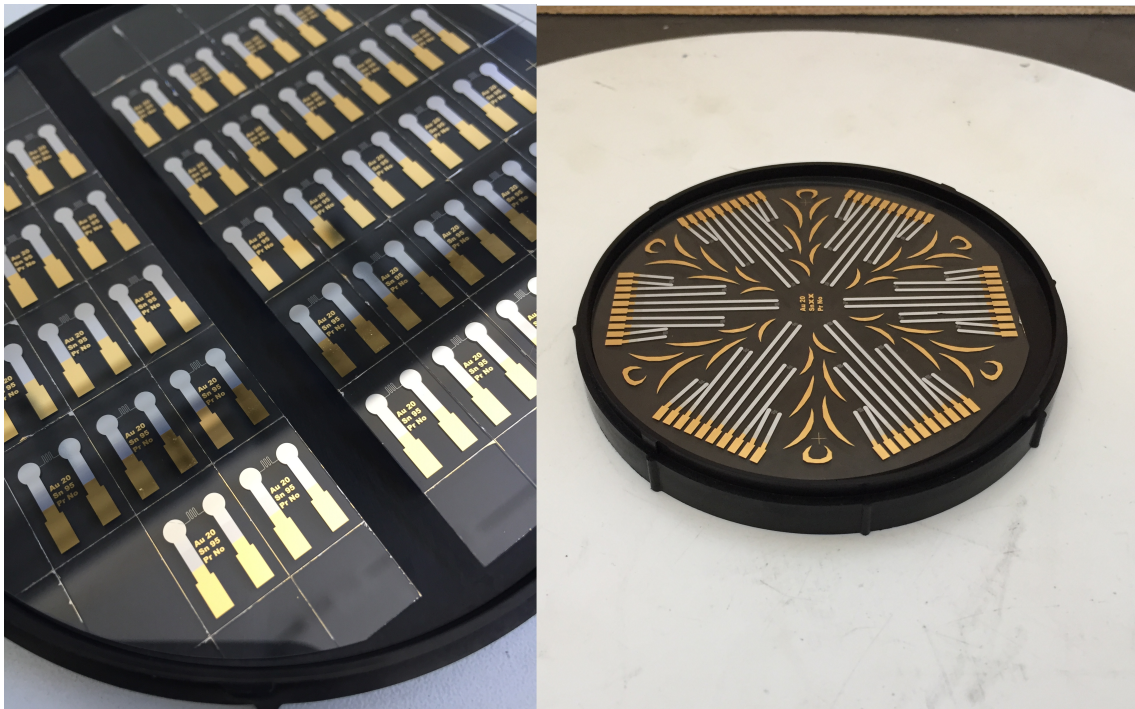


Figure 3.2: Picture of a prediced wafer with several Chip-TES (left) and a HEX-TES (right).

200 exposes the drawn geometries to UV light by a laser-diode. Everywhere where the photoresist was exposed to the UV light, it became easily soluble to organic solvents.

This material property is then exploited in the next step of the process, the developing. This was also done in the ACS 200, where the wafer was rinsed with an organic solution (AZ[®] 726 MF, MicroChemicals GmbH) for 30 seconds, then washed with ultrapure distilled water and finally hard baked at 100 °C for two minutes. While the organic solvent flows around the wafer, all the exposed photoresist and its underlying lift-off remover dissolves, leaving gaps on the surface of the wafer in the form of the desired pattern. The heating of the residual polymer slightly dehydrates and therefore stabilises it.

3.1.2 Metallisation

The actual thin films were fabricated by metal evaporation in an Alcatel EVA 600 evaporator. This evaporator can evaporate metals by Joule effect heating (e.g. for Au and Sn) or by the application of an electron beam (e.g. for Ti).

The wafers are placed in the vacuum chamber of the evaporator, on a rotating substrate holder facing towards the evaporation sources. A shutter can be interposed to stop the flow of vapour from the sources at the bottom towards the wafers at the top. The vacuum needed for the process is given by a pumping circuit with a turbomolecular pump type Alcatel 5900 CP, with which a pressure as low as 5.5×10^{-7} mbar can be reached.

The evaporation rate and the overall deposited amount of metal was measured by a SyconTM STM-100 thickness monitor. This detection method monitors the resonance frequency of a 6 MHz oscillating quartz crystal. When evaporated metal atoms deposit on the crystal surface, the resonance frequency of the crystal is shifted due to the increase of its mass by the deposited metal. The relationship between the frequency change and the mass of the metal film is known and thus the thickness of the deposited film not only on the crystal but also on the wafers can be calculated [84]. The input for the calculation are material dependent parameters as well as the so-called tooling-factor, which is a geometry dependent proportionality constant between the thickness of the deposited film on the crystal and that on the substrates.

The current of the electron gun or Joule heating boats was increased to preheat the metal source, and finally to evaporate it. When the conditions were appropriate (stable evaporation rate) the shutter was opened, and the thickness and deposition rate were monitored cautiously. When the desired thickness was achieved, the shutter was closed to stop the flow of vapour towards the wafers. This was done in the same fashion for the three metals used in this work.

During the metallisation stage, the thin film covers fully the wafer. On the zones that had been developed earlier, the metal is directly deposited on the glass, while everywhere else, it rests on a polymer layer.

3.1.3 The lift-off

The last step of the production consists of removing the remaining polymer layer with metal on top, to keep the metal only on the developed zones. The substrates were put

Table 3.1: Details of the TES fabricated at CMI.

Name	Date produced	Thickness Au layer (nm)	Thickness Sn layer (nm)
Au20Sn125	26.07.2017	20	125
Au20Sn150	19.07.2017	20	150
Au20Sn175	20.07.2017	20	175
Au20Sn200	21.07.2017	20	200
Au20Sn225N1	24.07.2017	20	225
Au20Sn250	25.07.2017	20	250
Au20Sn212	24.08.2017	20	212
Au20Sn225N2	28.08.2017	20	225
Au20Sn237	25.08.2017	20	237

in a bath of MICROPOSITTM Remover 1165 (mixture of several organic solvents, Dow Chemicals). The organic solution dissolves the photoresist as well as the LOR 5A, removing also the attached metal layer. This process is therefore called lift-off. Only the metal which was directly deposited on the glass stays on the surface, leaving the desired geometries of metal on the substrate. The use of a double polymer layer accelerates the lift-off, as the LOR works as a soluble sacrificial material. A more detailed description of the working principle can be found in [85]. After the lift-off bath, the wafers were rinsed in isopropyl alcohol to remove traces of the MICROPOSITTM Remover 1165, washed in distilled water and finally dried with a compressed air gun.

It is important to mention that after depositing the superconducting thin film, particular care was taken to avoid any exposure to heat.

3.1.4 The produced sensors

The sensors material consist of a metallic bilayer of 20 nm of gold followed by a layer of tin whose thickness was varied. An overview of all fabricated compositions is given in Table 3.1.

Since the ratio of the deposited metals is crucial for the performance of the sensor, a determination of the tooling factor of the thickness measurement device was done systematically for each evaporation source at the beginning of the production series.

For this, clean wafers were partly covered with stripes of Kapton[®] (DuPont). A layer of the metal was deposited on the wafers. By removing the Kapton[®] film, small stripes of the metal films were created. The thickness of the layers was then determined by measuring the step between the surface of the wafer and the metal film with a profilometer (see Section 3.2.1). Based on this measurements, the tooling factor for each of the metals was calculated.

3.2 Metallographic studies

3.2.1 Profilometry

Profilometer measurements of the thin films were conducted at CERN to calibrate the evaporation thickness monitor. A stylus profilometer (Bruker Dektak XT) was used. The measurement range was set to 6500 nm and a 12.5 μm stylus type was chosen. To determine the thickness of the film, the step between the wafer and the metal film was measured. The scanning length was between 1500 μm and 3000 μm , depending on the width of the measured stripe. The moving speed of the stylus was 0.05 mm s^{-1} and a force of 3 mg was applied. The measurement for a single sensor was at least repeated three times. For more detail on a stylus profilometer, see Appendix B.1.

3.2.2 Scanning electron microscopy

A Zeiss XB540 Focused Ion Beam device together with a Secondary Electron Secondary Ion Detector was used to analyse the microstructure of the thin films by scanning electron microscopy (SEM). To obtain conducting samples, copper tape was glued on the pads of the sensors with silver paste and connected to the metal support of the microscope. To reduce charging effects of the borosilicate glass during the microscopy, the sensors were coated with a 5 nm thick layer of graphite. The vacuum inside the microscope during the examination was approximately 1×10^{-3} mbar. The accelerating voltage of the scanning electron microscope was 10 keV. The working principle of SEM is explained in Appendix B.2. Post processing of the images was done with the open source software “ImageJ” [86] to measure distances and areas on the picture. By defining the length of the scale bar in pixels, the measured distances on the image can be transformed to the real values.

3.2.3 Energy-dispersive X-ray spectroscopy

The chemical composition of the films was investigated by energy-dispersive X-ray spectroscopy (EDS). The analyses were conducted in the same setup as the SEM. An Oxford Instruments Silicon Drift Detector was used for measuring the emitted X-rays. Further information about the theoretical background of EDS can be found in Appendix B.3. EDS analyses of the complete area shown on the image as well as on specific selected spots were conducted. The resulting compositions were calculated in mass and atomic fraction.

3.3 Heat treatment

Several samples of Chip-TES with the composition AuSn225N2 have been annealed to study the influence of heat treatment on the performance of the TES. This was done in an annealing oven (Cartridges Vertical Annealing System - VAS) at CERN under argon atmosphere (impurities < 10 ppm). The pressure was controlled with an oil diffusion HV-pump. The samples were annealed at 52 °C or 120 °C for 10 minutes. To control the annealing temperature and prevent overshooting, a moderate ramp rise in temperature of 50 K h^{-1} was chosen. During the annealing process, chamber pressure as well as the sample temperature are recorded. The heating is regulated by comparing the furnace temperature

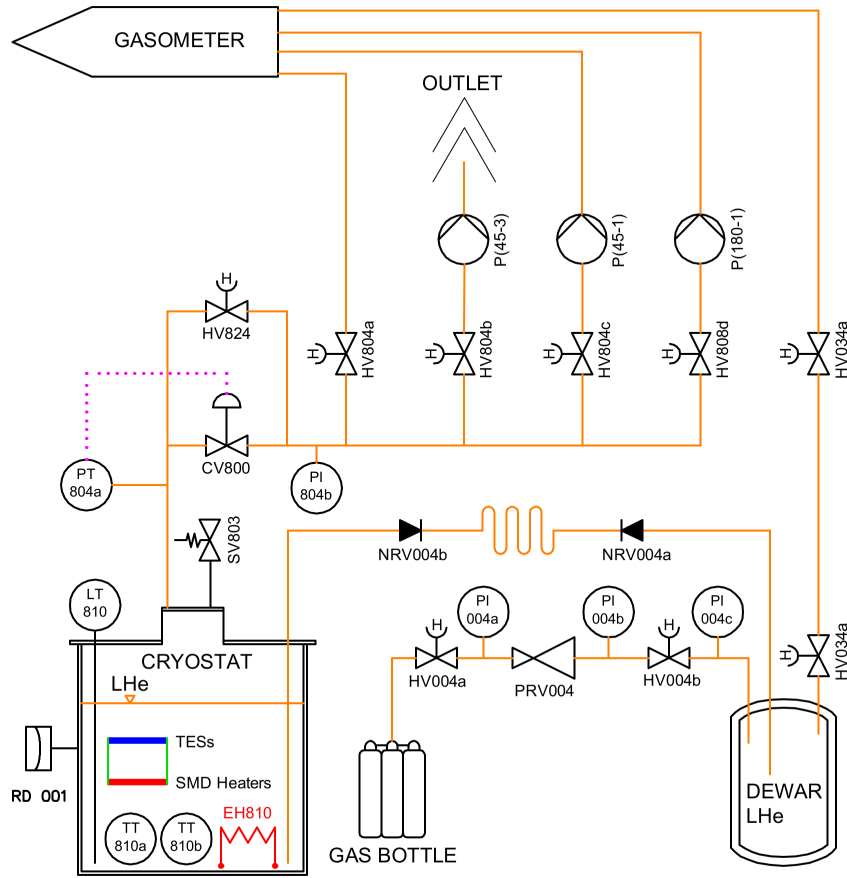


Figure 3.3: Simplified P&ID of the cryogenic testing setup at CERN from [6].

measured by a thermocouple with the actual set point. Since sample and oven temperature are not necessarily the same, test runs with dummy samples were conducted to see the relationship between the thermocouple and the actual temperature. Based on these results, a set point with the desired temperature curve was created. Some of the annealed samples were afterwards metallographically studied and others were tested in the cryogenic setup.

3.4 Cryogenic testing facility

The cryogenic experiments were carried out in the Central Cryogenic Laboratory of CERN. Piping, instrumentation system and cryostat were provided by the laboratory as well as the data acquisition system and the cabling [6].

A simplified P&ID of the setup is shown in Fig. 3.3. The cylindrical cryostat (Wessington Cryogenics Ltd) has an inner diameter of 200 mm and 1200 mm in height. The insulation vacuum in the double wall was approximately 1×10^{-5} mbar at room temperature and 1×10^{-7} mbar at 4.2 K. Prior to the cryogenic test, the support with the desired testing configuration, a resistance heater EH810, the temperature sensors TT810a (Pt 100) and TT810b (CernoxTM1050), the level gauge LT810 (HLG210, Cryogenics Ltd) and the stand

pipe were mounted inside the cryostat and connected through the closing top flange to the outside.

3.4.1 Cryostat purging

After closing the cryostat, it was purged to minimize residual gases which could freeze during the cooldown. This was done by opening the valve (HV804b) to the vacuum pump P(45-3) which is connected to an outlet. When the maximum vacuum was achieved, the valve was closed and the valve (HV804a) to the gasometer was opened. The gasometer represents the helium recuperation system of the laboratory. It connects all the facilities to a helium liquefier. The helium gas inside the gasometer is always at a slight overpressure. This helium gas was then used to fill up the cryostat. This cycle (pumping and refilling) was repeated five times.

3.4.2 The cooldown

To cool down the system, a liquid helium transfer was done. The liquid helium was stored inside a high quality dewar, filled at the laboratory liquefier. The Dewar was connected to the cryostat by a vacuum-insulated transfer line, which was plugged into the standpipe. The supply dewar was pressurised to 150 mbar reference pressure (PRV004) with a helium gas bottle (purity > 99.999%) to force a steady flow of liquid helium. The temperature inside the cryostat during the cool down was monitored by temperature sensor TT810a. When the boiling point of helium was reached, the level gauge was turned on to check the liquid helium inside. As soon as a filling height of 1000 mm was achieved, the transfer was stopped by reducing the pressure in the dewar first, and then disconnecting the transfer line. The valves NRV004a and NRV004b close automatically when no transfer line is plugged in.

3.4.3 Temperature control in He-II regime

After the previous steps the cryostat was full of liquid helium at 4.2 K and near atmospheric pressure. To achieve the conditions necessary to have saturated helium II, the pressure inside the cryostat had to be decreased. This was done by closing the valve HV804a, to isolate the system from the gasometer, and opening the valve (HV804c) to the vacuum pump P(45-1).

The pressure was monitored by a manometer (PT 804b). When the pressure was low enough, the by-pass (HV824) was closed. This allows a controlled regulation of the pressure inside the cryostat by the use of valve CV800 (which had been closed so far). For a desired temperature, the corresponding pressure value (see Appendix A.3) was given as target value to an MKS Type 651C pressure controller system. This regulation system acts on valve CV800 to regulate the pressure inside the cryostat as measured by the MKS pressure sensor PT804a. If temperatures below 1.7 K were needed, instead of P(45-1) the more powerful vacuum pump P(180-1) was used.

By monitoring the fluid level with LT810 during the tests, it was verified that the mounted testing setup was completely covered in liquid helium.

3.4.4 Heating of the cryostat

After the measurement in He-II was completed, the system was heated up to 4.2 K (at 1 bar) by an electric resistance heater EH810, with all valves HV804 closed. When the pressure was 1 bar, valve HV804a was opened and the heater EH810 was kept on till the whole setup reached room temperature.

3.5 Testing of TES

3.5.1 Experimental setup

The Chip-TES ($\text{Au}_{20}\text{Sn}_{225}\text{N}_1$) were mounted in a measurement setup, as the one shown in Fig. 3.4. The mechanical support was made out of a glass fiber epoxy laminate. The sensors were fixed with spring-connectors on the sensor plate for electrical connection. The spring-connectors were linked to a 4-pin connector glued on the bigger holder element.

To produce second sound pulses, 9 SMD heaters ($6\text{ mm} \times 3\text{ mm}$) with a resistance of $47\ \Omega$ were placed on a polymer plate, in a row. The distance between the heaters was 10 mm. The heater plate was mounted facing the TES (see Fig. 3.5) at a distance of roughly 50 mm from the sensors.

3.5.2 Signal acquisition and heater control

The electrical signals of the TES were delivered from the 4-pin connectors to the outside of the cryostat by ManganinTM cables going through the feedthrough and a patch panel. The wires were connected via a 4-wire sensing technique to prevent errors in the measurements due to the resistance of the cables (see Appendix C.1). A KeithleyTM 6220 Programmable Current Source was used to deliver electrical current to the TES. NI 9251 analogue input modules were used to measure the voltage. The modules are assembled in a NI CompactDAQ 8-Slot chassis, which is connected to the measurement computer via USB. A LabVIEW interface was used to record bias current and voltage of each sensor.

The NI modules for voltage recording have very high sampling rate (up to 100 kS/s) and high resolution (24 bits). This has been a requirement in order to record the very fast and small signals induced by second sound on the sensors (see Section 4.2).

To produce second sound waves out of the SMD heaters, square current pulses were generated by triggering the heater. The heater circuit was fed with a programmable power supply (HM8142, HAMEG Instruments) or rechargeable 6 V batteries (RS Pro) wired in series. A NI 9472 module was used as an electronic relay, controlled by the LabVIEW interface. The duration of the pulse can be set as an integer number of milliseconds. The current on the heater was also recorded by measuring the voltage drop on a $0.6\ \Omega$ resistance in series.

3.5.3 Superconducting transition of TES

The characterisation of the sensors was done by recording the voltage $V(I, T)$ on the TES while sweeping the bias current I in 20 μA stages at stable bath temperature T . The range of the current sweep was from 0 mA to 4 mA. This experiment was repeated at different stable bath temperatures between 1.5 K and 2.1 K in steps of 50 mK.

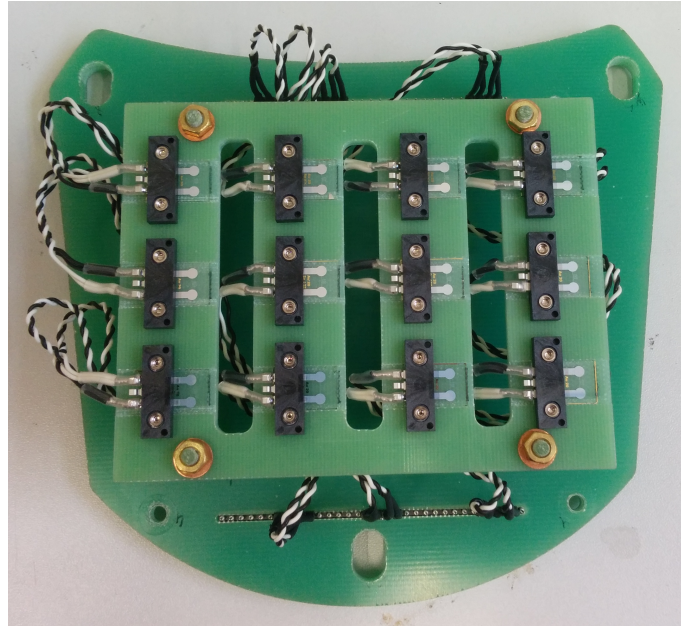


Figure 3.4: Assembled measurement setup for the characterisation of 12 Chip-TES on a G10 support. For better perspective, the sensor plate was mounted facing in the opposite direction than normally during testing.

3.5.4 Second sound detection with TES

Experiments were conducted to evaluate the capability of TES as second sound detectors. The bath temperature was stabilised at $T = 1.8\text{ K}$. The sensors were biased at constant current I , recording the signal $V(t)$ when pulsing on a heater. Second sound pulses were produced with the SMD heaters with the triggering option of the LabVIEW program. The voltage was recorded after triggering with a sample rate of 20 kHz . The electric pulses on the heaters had typically an amplitude of 30 V and a duration of 1 ms , but these values could be changed whenever the effect of these parameters was the interest of study.

The power P emitted by the heater can be calculated by a combination of Joule's and Ohm's law:

$$P = VI = \frac{V^2}{R} . \quad (3.1)$$

Following this, the heat flux $\dot{q}$ of a heater with an area A is given as

$$\dot{q} = \frac{P}{A} = \frac{V^2}{RA} . \quad (3.2)$$

3.6 Angular dependence of second sound

3.6.1 Sensors and heaters setup

In order to investigate the spherical propagation of second sound, a different setup shown in Fig. 3.6 was created. On a circular holder element, four support plates were fixed. On each plate, an OST and a TES were positioned. The holder has a hole that allows the stand pipe

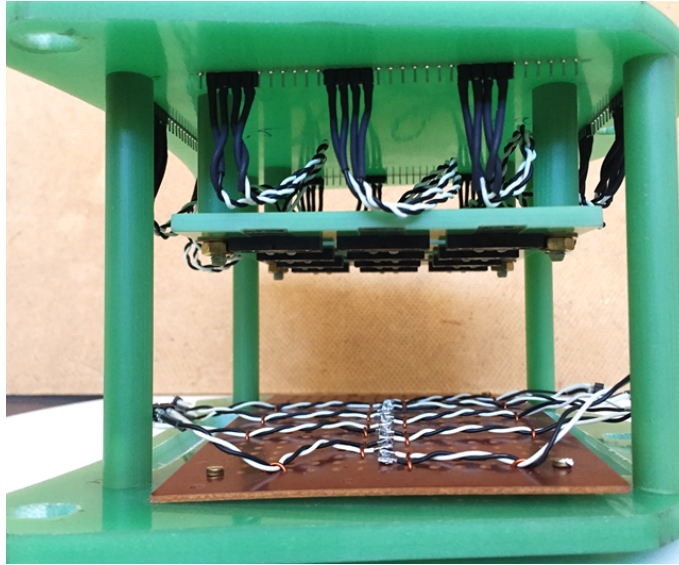


Figure 3.5: Assembled measurement setup for the second sound detection. 9 SMD heaters are mounted on a polymer plate on the bottom, facing the TES (only the black spring connectors are visible in this perspective).

to pass through. The plates were positioned so that the sensors are in a plane perpendicular to the direct line of sight joining the standpipe and the sensors. The angle between the successive lines of sight was 30° . On a stainless steel holder connected to the standpipe, four SMD heaters were glued on one of its walls. Two smaller heaters ($6\text{ mm} \times 3\text{ mm}$, $30\ \Omega$) were positioned in such a way, that one was exactly on the height of the OST centre while the other one was directly in front of the TES. Two bigger heaters ($7\text{ mm} \times 8\text{ mm}$, $33\ \Omega$) were glued 25 mm above a small one, respectively. Thereby, the big heaters can be positioned in front of the sensors by pushing the standpipe 25 mm down. The orientation of the heaters was changed by rotating the standpipe. To be able to measure the angle outside the cryostat, a metallic plate with graved lines displaying the angle was mounted on top of the cryostat flange. A small blade hanging from the outer part of the standpipe indicated the current angle β .

Since all the sensors are placed symmetrically on a circumference with a 30° angle to each other, the angle between each sensor and the heater front α_i could be calculated on the base of β . This is schematically displayed in Fig. 3.7.

3.6.2 Signal reading and heater control

The connecting, controlling and recording method for the TES signal and heaters was exactly the same as in Section 3.5.2.

The OST were connected with coaxial cables to a filtering/amplifying box outside the cryostat to increase the signal and reduce noise. The circuit layout of the amplifiers can be seen in Appendix C.3. The amplifiers were connected to a digital oscilloscope (DPO 3014, Tektronix) to read out the processed signal with a sampling rate of 250 kHz. To charge the OST, the programmable power supply (HM8142, HAMEG Instruments) with a constant voltage of 30 V was used. The heat pulses were generated by four rechargeable 6 V batteries

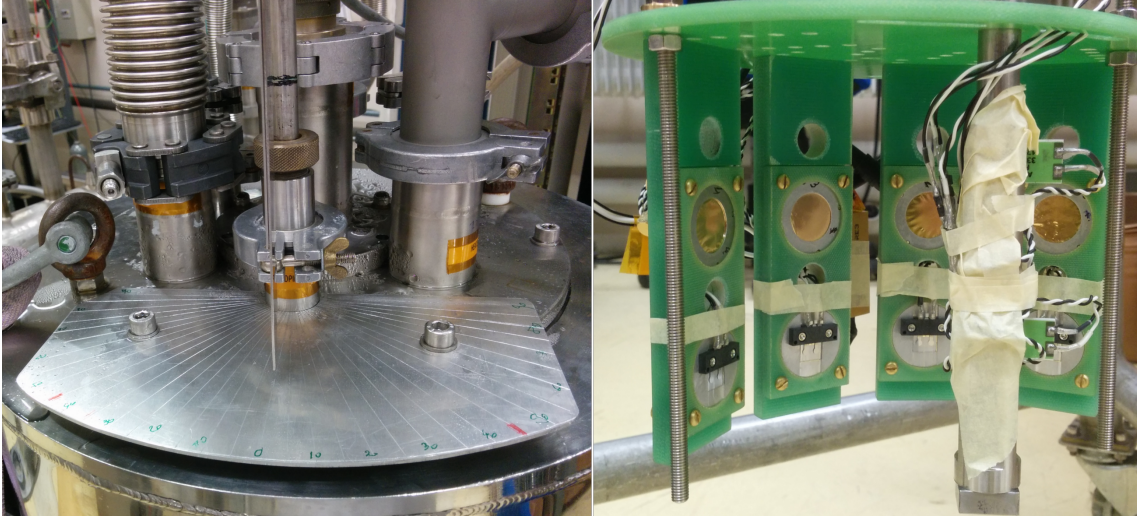


Figure 3.6: Left: Metal plate mounted on top of the cryostat with a blade hanging from the standpipe to indicate the current angle between the heaters and the sensors.

Right: Image of the setup which was mounted inside the cryostat. Four G10 supports, each with an OST (recognisable on the gold foil) and a TES are showing towards the heaters which are glued on the standpipe. From this perspective, only the back of the bigger heaters are visible. The duct tape was used to assure that no cables move in front of the heaters.

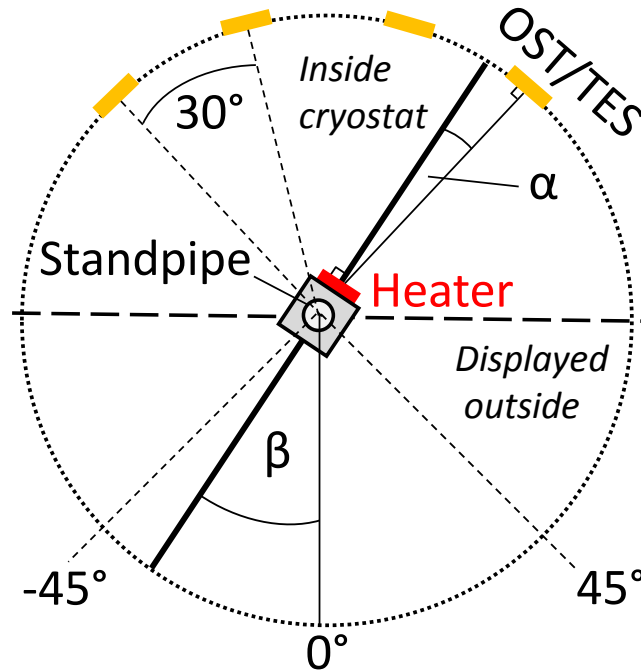


Figure 3.7: Sketch of the angle measurement setup showing the relationship between the angle β measured outside and the actual angle between the sensor and the heater front α . For $\beta = -45^\circ$, the heater faces TES/OST 1 perpendicular ($\alpha = 0^\circ$).

(RS Pro) wired in series instead. Finally it is important to mention that all devices were grounded to the same earth potential.

3.6.3 Measurement protocol

At stable bath temperature, heat pulses were generated on the small heater at the TES level, and the detection of second sound with the TES was recorded, similar to the experiment in Section 3.5.4. Afterwards, the standpipe was rotated by 5° , the new angle was noted and the experiment was repeated. The range of the measuring angle β was from -45° to 45° , limited by the cabling of the heaters. After the measurement run with the TES was completed, the same process was carried out, now detecting the second sound with OST and pulsing on the other heater.

To study the influence of the heater size, the bigger heaters were placed in front of the sensors by lowering the stand pipe and the full process repeated. The support plates of the sensors were moved at different distances to the heater while keeping the angle constant. By this, the distance dependence of the signal could be investigated. To study the influence of the heater size, the bigger heaters were placed in front of the sensors by lowering the stand pipe and the full process repeated. When desired, the heat flux could be varied by removing a battery from the supply circuit of the heater.

3.7 Computational data processing

Due to the amount of experiments, the recorded signals had to be analysed computationally to be able to calculate the time of flight of the second sound waves for each measurement.

In case of the TES, the time stamp of the activated trigger was detected by identifying the voltage increase of the resistor which was connected in series with the heater. The unperturbed signal was characterised by calculating its average and its noise level. The calculation was based on values recorded approximately 10 ms before the trigger was activated. The programme searched for the maximum value of the sensor exceeding the average plus the noise. To accelerate this process, the programme looked only for the maximum within a given interval. Outside this interval, a detection of second sound would be not physically plausible due to the location of the sensors with respect to the heater.

Once the maximum was detected, the programme searched for the start of the peak, which represents the arriving time of the second sound wave at the sensor. This was done by looking for the last recorded point before the detected maximum, which was below the average plus noise values before the trigger.

The algorithm for the OST was similar to that of the TES. However, since the arrival of second sound decreases the voltage measured on an OST (see Section 2.4.2), instead of a maximum the programme looked for a minimum. The arriving time of second sound on the OST was determined by looking for the last recorded value before the detected minimum, which was still above the pre-trigger average minus noise level. Since the trigger system could be connected directly to the Oscilloscope, the time stamp of the start of the heat pulse was recorded automatically when activating the trigger.

3.8 Analysis of measurement uncertainty

To evaluate the uncertainty of measurement of the investigated parameters in this thesis, the systematic approach given by the “guide to the expression of uncertainty in measurement” (GUM) [87] was used.

The experimental data of second sound measurements were taken by repeating the experiment under the same conditions. The measured parameters were the time of flight of the second sound TOF and the voltage on the sensor V_{2s} . Thus, an evaluation “Type A” was done in order to determine the arithmetic mean $\bar{q}$ for the 5 independent observations q_k as well as the experimental variance of the observations $s^2(q_k)$

$$\bar{q} = \frac{1}{n} \sum_{i=1}^n q_k \quad ; \quad s^2(q_k) = \frac{1}{n-1} \sum_{j=1}^n (q_j - \bar{q})^2 . \quad (3.3)$$

The Type A standard uncertainty is defined as

$$u_A = s(\bar{q}) = \sqrt{\frac{s^2(q_k)}{n}} . \quad (3.4)$$

It is important to mention that only in this section u is referred as the uncertainty, whereas in all other sections it represents the second sound velocity u_2 . With these concepts, the corresponding standard uncertainties u_τ and $u_{V_{2s}}$ were calculated.

Although the low amount of repetitions, Type A evaluation (GUM 4.2) was chosen, because it was difficult to find an assumption for the uncertainty of the computational analysis for the second sound detection. The same applies for the electrical noise, which intensity can not be simple explained by the measurement uncertainty of the current source. It is important to mention that for the construction of confidence intervals, due to the low number of observations, the t -distribution has to be taken into account (GUM G.2). For a degree of freedom of 4, the type A standard uncertainty has to be multiplied with a factor of 1.14 or 2.87 to get a confidential level of 68.27 % (k-factor = 1) or 95.45 % (k-factor = 2), respectively. Unless specified differently, all given uncertainties in this work are calculated with a coverage factor $k = 2$. Also the uncertainty of the thickness of the films measured by the profilometer was estimated by this method.

However, the transition curves of the TES were measured only once. Also the uncertainty of the measured distance and angle between heater and sensor can not be estimated with the method described above. In this case a type B evaluation of standard uncertainty (GUM 4.3) was performed. A table with all measurement uncertainties of the devices used in this work can be found in Appendix D.1.

For the measurand where no uncertainty values were given, the Type B standard uncertainty u_B , based on a rectangular distribution, had to be estimated by

$$u_B(x_i) = \frac{a}{\sqrt{3}} , \quad (3.5)$$

where x_i is the measured estimate of the true measurand X_i and a half the interval

given by an estimated upper and lower bound. Additionally, the uncertainty of the second sound velocity taken from the literature has to be considered. The resulting u_B are also summarised in D.1.

The final true output value is often a function of several input quantities

$Y = f(X_1, X_2, \dots, X_N)$. Therefore, the propagation of the uncertainty of each of these input quantities $u(x_i)$ has to be considered. Assuming that the input quantities are not correlated, the combined standard uncertainty $u_c(y)$ (GUM 5.1) of an output estimate y can be calculated:

$$u_c(y) = \sqrt{\sum_{i=1}^N \left(\frac{\partial f}{\partial x_i} \right)^2 u^2(x_i)} . \quad (3.6)$$

The evaluated parameters of the present work, as well as their input parameters and the corresponding combined standard measurement uncertainties are listed in Appendix D.2.

4. Results

4.1 TES characterisation

4.1.1 Influence of microstructure on superconductivity

Thickness measurements

It has already been pointed out in this thesis that the ratio between the normal conducting gold and the superconducting tin of the film is crucial for its performance. Since already Kovács [6] reported problems with the reproducibility of the results of Borner *et al.* [75], the thickness of each sensor was measured by a stylus profilometer to double check the amount of deposited metal on the substrate. The results are listed in Table 4.1. The name of each sensor indicates its composition and if needed information about the production series. It can be seen that the first six compositions deposited only show aberrations below 10 % to the expected values. All sensors measure a thickness a few nanometres above the expected value based on the thickness of each metal deposited. This can be explained by the formation of new phases of gold-tin alloys (see Appendix A.2) during the evaporation process which have different densities than the pure metals [88].

One month after the first fabrication, a second production series of TES at CMi was done in order to verify the reproducibility of the evaporation process. However, as displayed in Table 4.1, the resulting films are more than 15 % thicker than expected, although an additional calibration as described in Section 3.1 was done again.

As the results for the first production series match the expectations much better, they are considered to reproduce accurately the desired composition. Therefore, almost all of the following experiments were conducted with TES from this fabrication series.

Analysis of microstructure

According to Borner *et al.* [75] the microstructure plays an important role in the superconducting behaviour of the metallic bilayer. That is why SEM of the manufactured thin films were performed. In Fig. 4.1 SEM images of the sensing stripes of TES with a composition with 20 nm of gold and 150 nm, 200 nm, 225 nm and 250 nm of tin are shown. In all pictures an heterogeneous structure is observed, similar to the one reported by Borner [75]. Grain-like features with higher contrast (bright) are densely spread over a uniform background layer with lower electron contrast. Measuring the chemical composition of these two phases for the Au20Sn225 with EDS shows that the features with an approximately atomic ratio $n_{Sn}/n_{Au} = 7.15$ are very rich in tin compared to the background, where the ratio is roughly three times lower ($n_{Sn}/n_{Au} = 2.56$). Other elements detected in considerable amount are oxygen and silicon, the main components of the borosilicate glass.

Although the general structure is the same, there are significant differences between the images with different compositions when it comes to the features itself. Especially the film

Table 4.1: Comparison between the expected thickness of the TES and the actual thickness measured with a profilometer.

Name	Expected Thickness (nm)	Measured Thickness (nm)	Deviation (%)
Au20Sn125	145	149.7 ± 5.2	3.14
Au20Sn150	170	174.2 ± 9.0	2.41
Au20Sn175	195	200.3 ± 6.8	2.64
Au20Sn200	220	231.0 ± 8.0	4.76
Au20Sn225N1	245	252.0 ± 9.0	2.78
Au20Sn250	270	289.6 ± 7.8	6.78
Au20Sn212*	232	282.8 ± 10.4	17.96
Au20Sn225N2*	245	310.3 ± 6.0	21.04
Au20Sn237*	257	314.8 ± 9.8	18.36

*produced one month later

with only 150 nm Sn stands out. Black spots inside the features can be recognised. The amount of silicon and oxygen of these black spots measured by EDS is almost 34 % higher. This means that much more electrons of the substrate have been excited, indicating that the film is much thinner on these locations.

All the images are displayed in the same scale, so that the size of the features can be compared. A trend of growing size and decreasing spacing between the features with increasing tin content is clearly visible. This observation was confirmed by estimating the Sauter mean diameter of the features on the images with the open source image processing program “ImageJ” [84, 89]. The features of the Au20Sn150 measure between 200 and 600 nm in size, while the sensor with 200 nm of Sn has features with a size of 220 to around 900 nm. The size of the grain-like features of the Au20Sn225N1 lies in the range of 400 nm to 1000 nm, whereas the features of the film with a deposited 250 nm thin film of tin has even features with a size between 400 nm and 1270 nm. However it has to be mentioned that these values are only an approximation for a qualitative interpretation. For a quantitative analysis, much more images have to be analysed in order to get a statistically significant result.

In Table 4.2 the results of the EDS analyses measured over the area shown in the SEM images are listed. With increasing amount of tin, the mass fraction of the tin rises, too, as expected. Comparing the values reveals that a linear increase of the thickness of the tin layer by 25 nm leads to a corresponding raise of around 3 at%. This confirms that the deposited thickness for the first production series was correct.

Superconducting transitions of the sensors

Fig. 4.2(A1), (B1) and (C1) show the resulting V-I curves measured for the sensors Au20Sn200, Au20Sn225N1 and Au20Sn250 by the method explained in Section 3.5.3. All other sensors with lower amount of tin did not show any superconducting behaviour, even

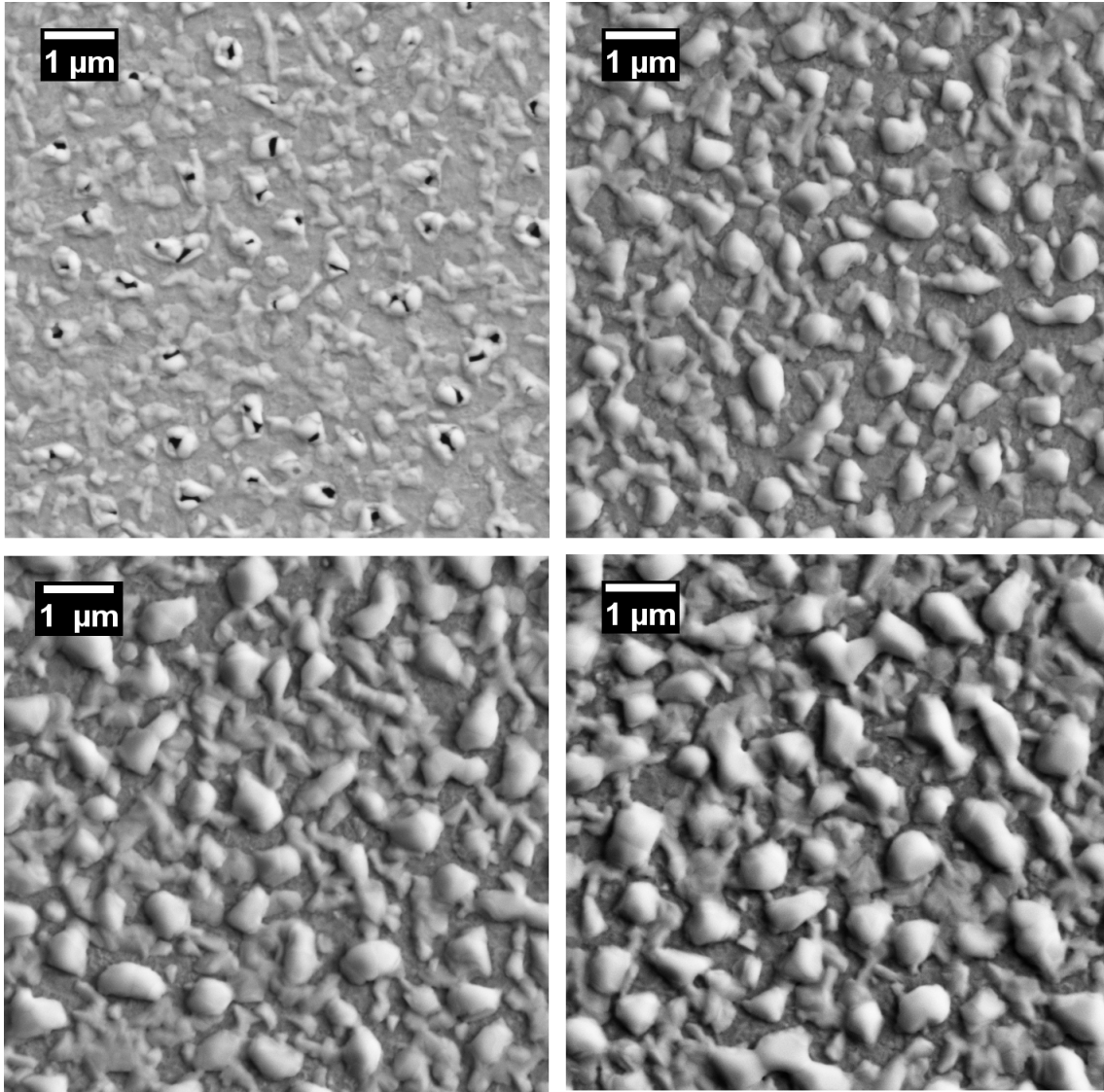


Figure 4.1: SEM images of the surface of sensors with different composition showing the microstructure of the thin film. Top: Au20Sn150 (left) and Au20Sn200 (right); bottom: Au20Sn225 (left) and Au20Sn250 (right); The uniform background layer and grain-like features with higher electron contrast are clearly distinguishable, resulting in a heterogeneous structure. With increasing amount of tin, the features increase in size and the spacing between them reduces.

Table 4.2: Results of the overall EDS analyses of the films for gold and tin.

Name	wt% Au	wt% Sn	at% Au	at% Sn
Au20Sn150	43.27	56.73	31.48	68.52
Au20Sn175	39.39	60.61	28.14	71.86
Au20Sn200	36.25	63.75	25.52	74.48
Au20Sn225N1	33.12	66.88	22.98	77.01
Au20Sn250	30.22	69.78	20.69	79.31

at the lowest temperature measured ($T = 1.5$ K). To obtain a better overview, only the curves at three different temperatures $T = 1.6$ K, 1.8 K and 2.0 K are displayed.

The curves of the sensor with 200 nm tin (see Fig. 4.2(A1)) show for the two higher temperatures a very linear behaviour. Only at 1.6 K for a bias current below 1.0 mA to 1.5 mA is the curve not a straight line, but shows a lower slope before matching the behaviour of the other two. This indicates that at low current the sensor is not resistive, producing zero voltage drop, and at a given point goes gradually through the transition from super- to normal conducting. At higher temperatures and higher bias currents, according to the slope, the measured voltage on the sensor follows Ohm's law, with the slope being its normal resistance.

In Fig. 4.2(B2) and Fig. 4.2(C2) the S-shape of the curves is more pronounced. The fast increase of resistance due to the transition leads to an abrupt raise in voltage over a range of a tens of μ A. By comparing the three figures, two trends can be observed. Firstly, with decreasing temperature, the transition region gets shifted to higher bias currents and the slope becomes steeper. This behaviour was expected, because a lower temperature allows higher critical currents for the superconductor, as it was explained in Section 2.1. The second trend observed shows the influence of the proportion of the film. With an increasing amount of tin the transition behaviour gets closer to that of the pure superconductor. This relation between the deposited amount of tin and the transition was also observed by Borner *et al.* [75]. However, Borner published his results for a TES with a layer of 100 nm Sn on top of a 20 nm Au layer on a quartz substrate, whereas in this thesis even for a sensor with layer of 175 nm tin no superconductivity has been recorded.

Thermometric performance of the sensors

In order to use the TES as thermometers, their thermal sensitivity $\partial V / \partial T$ has to be calculated. The sensitivity describes how much the voltage on the sensor changes with temperature at a certain bath temperature T while the bias current stays constant. In this thesis, the sensitivity of a TES biased with a current I was estimated numerically from the measured steady state data shown in the previous section, with the following finite difference formula:

$$\begin{aligned} \frac{\partial V}{\partial T}(I, T_j) \cong & \frac{T_j - T_{j-1}}{T_{j+1} - T_{j-1}} \frac{V(I, T_{j+1}) - V(I, T_j)}{T_{j+1} - T_{j-1}} \\ & + \frac{T_{j+1} - T_j}{T_{j+1} - T_{j-1}} \frac{V(I, T_j) - V(I, T_{j-1})}{T_{j+1} - T_{j-1}}, \end{aligned} \quad (4.1)$$

where V is the measured voltage, j indicates the value of temperature at which the calculation of sensitivity is done, and $j \pm 1$ is the next or previous value of temperature at which current sweeps were measured. Experiments conducted at CERN previous to this thesis have showed that steps of 50 mK are small enough to minimise the discrepancies due to the mathematical approximation of the deviation. Since the steps between the bath temperatures were not always exactly 50 mK, a weighting factor based on the model of the law of lever was used (the fractions which only include temperatures).

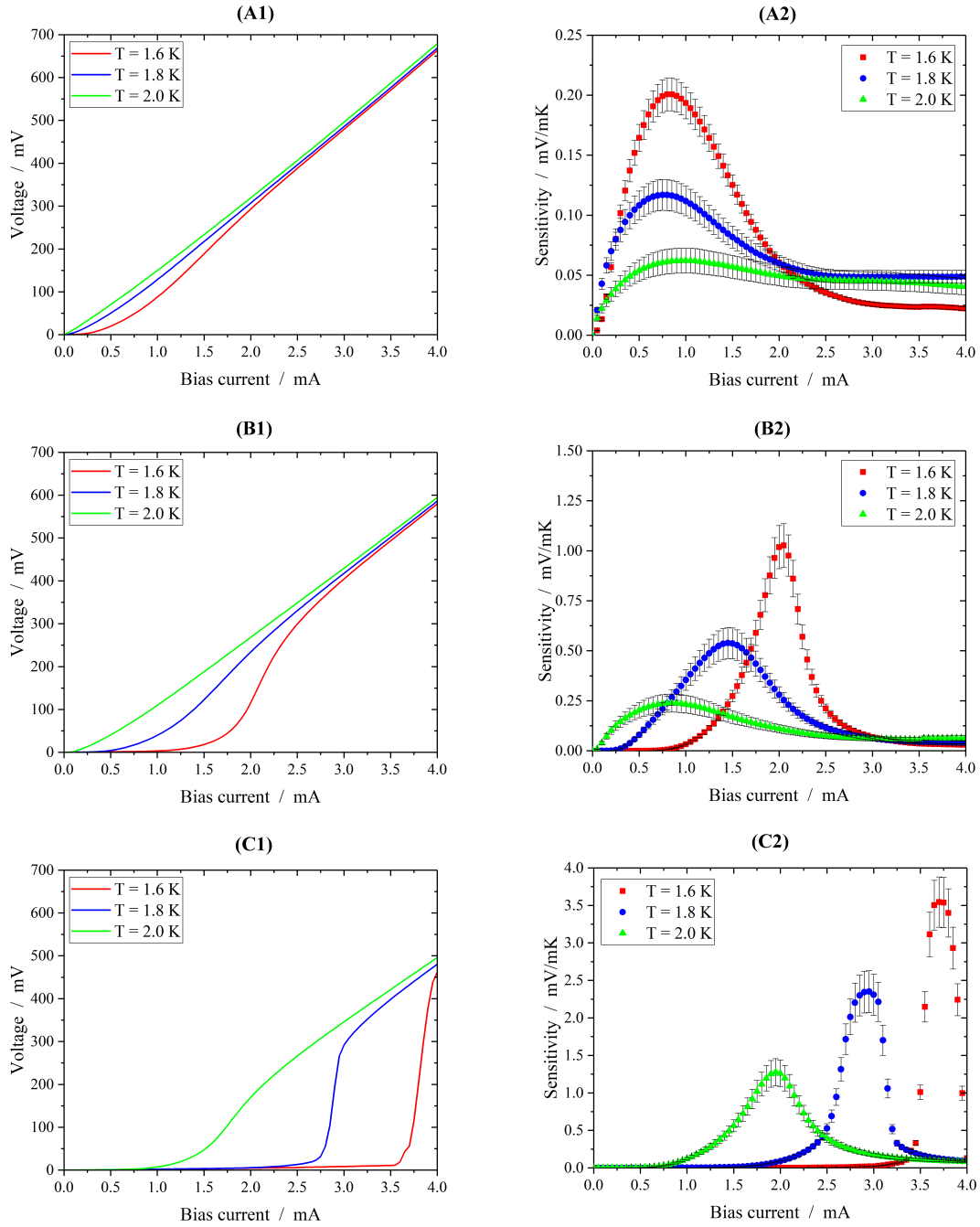


Figure 4.2: Top row: V-I curves (A1) and calculated sensitivity values (A2) for a Au20Sn200 TES at 1.6 K, 1.8 K and 2.0 K.

Middle Row: V-I curves (B1) and calculated sensitivity values (B2) for a Au20Sn225N1 TES at 1.6 K, 1.8 K and 2.0 K.

Bottom Row: V-I curves (C1) and calculated sensitivity values (C2) for a Au20Sn250 TES at 1.6 K, 1.8 K and 2.0 K.

The error bars for the voltage measurement lie within the plotted line and are therefore not shown.

In Figs. 4.2(A2)(B2)(C2) the corresponding sensitivity curves for the measured V-I curves are shown. As it was expected, the Au20Sn250 (C2) shows the highest sensitivity with a value of $(3.54 \pm 0.34) \text{ mV mK}^{-1}$ for a bias current of 3.70 mA at $T = 1.6 \text{ K}$ due to its sharp transition. Both, the maximum sensitivity and its corresponding bias current reduce with decreasing amount of tin. In accordance with that, sensor Au20Sn200 (A2) with the lowest tin ratio reaches even at 1.6 K only a sensitivity of $(0.20 \pm 0.01) \text{ mV mK}^{-1}$ when biased with a current of 0.80 mA. A conclusion which can be drawn from that is that at least a layer of 225 nm of tin (B2) should be deposited in order to achieve TES with a proper sensitivity for the classic temperature range of a cavity test (1.8 K-2.0 K).

In general, a sensitivity as high as possible is desired in order to obtain a clear signal. Hence, the Au20Sn250 should be superior to the Au20Sn225 due to its higher sensitivity. However, when biasing the Sn20Au250 with $I = 2.95 \text{ mA}$ at a bath temperature of 1.8 K, large background noise in the order of several hundred microvolts was observed, making it impossible to operate with this sensor at stable conditions. Although the exact reason for this behaviour is unknown, a plausible explanation could be given by Irwin and Hilton [65], who describe noise initiated by a positive “electrothermal feedback” (ETF) for current biased TES which can lead to unstable behaviour. Since a TES transduces temperature fluctuations into an electric current signal by its change of resistance, it also feeds back electric current into temperature by Joule power dissipation. This effect gets intensified with higher bias current and increasing sensitivity, and could therefore play a role for highly sensitive TES like the Au20Sn250.

Finally, the observations of the microstructure and the thermometric performance of the sensors can be linked. With increasing amount of tin, the tin rich grain-like features of the heterogeneous grain size and the spacing between them reduces. This also increases the superconducting behaviour of the sensor causing higher critical currents, critical temperatures and a steeper transition. The microstructure therefore works as an indicator for the probable performance of the TES.

4.1.2 Influence of heat treatment

Since the literature [78] reports a fast diffusion rate of gold into tin, the influence of heat treatment to the sensor performance needed to be investigated. The temperature profile of the sensors and other parameters measured during the annealing process can be found in Appendix E.1. Because the annealing system used in this thesis is not intended to operate at temperatures below 200°C , some previous experiments had to be conducted in order to find a proper setpoint to obtain approximately the temperature curves desired. Not to waste any useful TES, several Chip-TES of the type Au20Sn225N2 were used.

The first run at a temperature of 52°C does not change anything on the microstructure or performance of the sensor. This is contrary to the results of Borner *et al.* [75]. However, Borner annealed his samples under vacuum atmosphere, while the experiment in the current work were conducted with argon to be able to control the temperature properly. The second aspect which has to be considered is the fact that Borner does not mention which temperature ramp he used to reach the 52°C . Finally, the composition of Borner’s TES are

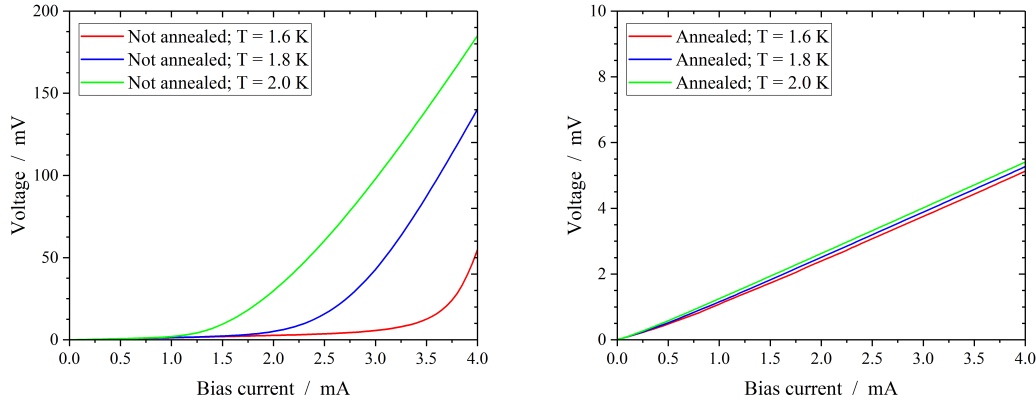


Figure 4.3: Measured voltage plotted over bias current for a Au₂₀Sn₂₂₅N₂ TES at three different temperatures, before (left) and after annealing (right). The annealed sensor behaves like a pure superconductor in the given bias current range. The small increase in voltage of approximately 5 mV can be explained by the resistance of the gold pads.

very different from the TES used in this work. All the things mentioned could affect the diffusion behaviour of the components within the TES and therefore explain the different results.

However, the results of the second run at a temperature of 120 °C are different. The V-I curves for a Au₂₀Sn₂₂₅N₂ recorded before and after the annealing process are shown in Fig. 4.3. The non-annealed sensor has a critical current between 1 mA and 3 mA, depending on the temperature. The fact that the curves do not overlap for the highest current measured, shows that the sensor is still in the transition zone and has not yet become fully normal conducting. Thus, this curves demonstrate that the Au₂₀Sn₂₂₅N₂ has a different composition and accordingly a different transition than the Au₂₀Sn₂₂₅N₁ (see Fig. 4.2 (B1)). The annealed sensor shows for all temperatures measured a purely superconducting behaviour. Even when biased with a current of 4 mA, the measured voltages are below 6 mV. The small increase of the voltage can be explained with the residual resistance of the contact pads, which consist of gold and titanium and hence are not superconducting above 1.6 K. This trend is completely the opposite to Borners results, who observed a reduction of the critical current when the sensor was annealed (see Section. 2.4.3, Fig. 2.18).

As it can be seen in Fig. 4.4, the microstructure of the sensor does not change during the annealing. This is not only contrary to Borners results, who observed the vanishing of the heterogeneous structure, but does also not fit with the conclusions drawn in the previous section. A change of the performance of the TES should be caused by a different microstructure.

Therefore, a second effect has to be taken into account. The results of the EDS measurements on the grain-like features and the background, listed in Table 4.3, could give an explanation. The spots where exactly the analyses was done are marked in Fig. 4.4. In the non-annealed TES, the features contain more than 94 % tin, whereas the background consists only of 58.42 % tin. In contrast, the two phases of the annealed wafer have almost the same

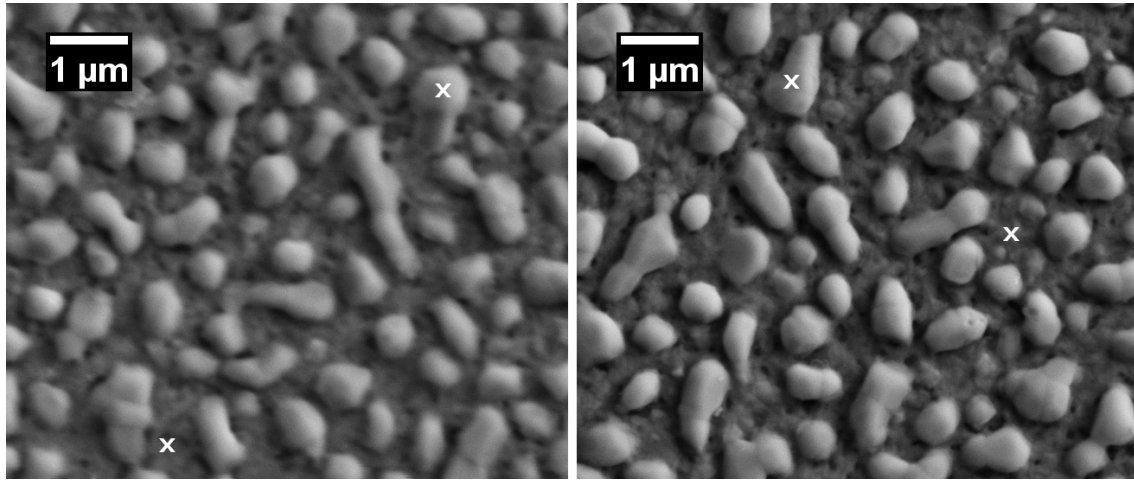


Figure 4.4: SEM images of the microstructure of a Au₂₀Sn₂₂₅N₂ TES without additional heat treatment (left) and after annealing it at 120 °C (right). The crosses mark the points where the composition of the film has been analysed by EDS.

Table 4.3: Results of the local EDS analyses of the annealed and non-annealed Au₂₀Sn₂₂₅N₂ for gold and tin.

Name	annealed	spot	at% Au	at% Sn
Au ₂₀ Sn ₂₂₅ N ₂	no	background	41.58	58.42
Au ₂₀ Sn ₂₂₅ N ₂	no	feature	5.68	94.32
Au ₂₀ Sn ₂₂₅ N ₂	yes	background	28.67	71.33
Au ₂₀ Sn ₂₂₅ N ₂	yes	feature	27.38	72.62

composition, only a difference of 1.29 % was measured. The most likely explanation for this vanishing gradient is the fast diffusion of gold atoms from the background into the features due to the higher temperature. The system moves towards its equilibrium state, where the distribution of the composition is uniform over the whole microstructure. This makes the film behave closer to a pure superconductor causing a better superconducting behaviour at higher bias currents as it was observed.

Another possible explanation could be that the annealing helps the material crystalline defects to heal and thus the superconductivity improves.

Finally it can be said that by annealing the TES, the critical current can be shifted to higher values. This could be helpful to “correct” TES with weak superconducting behaviour.

Since the test at 52 °C did not cause any changes on the TES, it can be assumed that storing the TES at room temperature should be sufficient to prevent a performance change due to interdiffusion. However, only one composition was tested, and it could be possible that sensors with a higher amount of gold diffuse already at lower temperatures. More experiments with different compositions, annealing temperatures and test duration are necessary to be able to determine the influence of the parameters precisely.

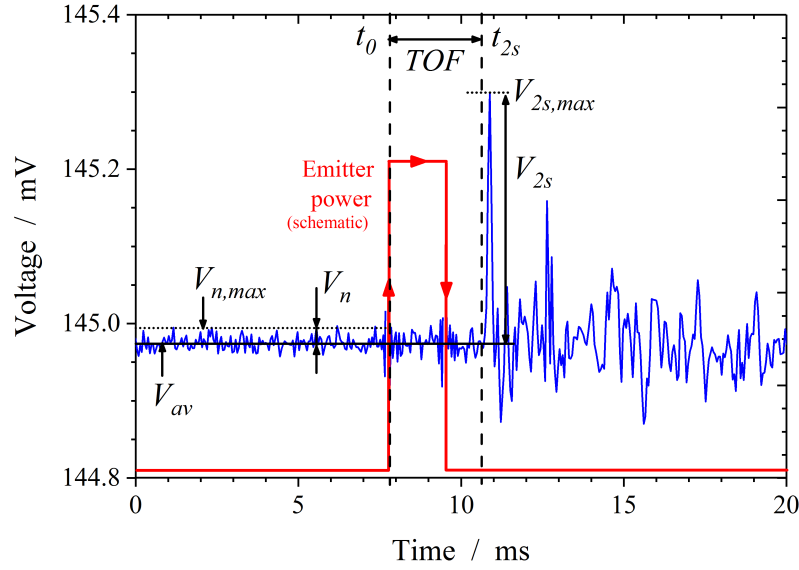


Figure 4.5: A characteristic second sound signal, detected with a Au20Sn225N1 TES at 1.8 K. The signal of the heater as well as the important parameters measured and calculated are displayed schematically to help clarify the evaluation of the measurement.

4.2 Second sound measurements

Through the procedure explained in Section 3.5.4, tests were conducted to evaluate the capability of the produced TES to detect second sound. The voltage drop on biased sensors was recorded while pulsing current on a heater. A characteristic signal, detected with a Au20Sn225N1 Chip-TES at 1.8 K is shown in Fig. 4.5. The signal of the heater is displayed schematically to help clarify the important measured values as well as the calculated results.

As a reference, the mean voltage on the sensor before the trigger V_{av} is calculated. To estimate the level of background noise, the measured maximum values of voltage before the trigger are averaged in $V_{n,max}$. The actual noise level V_n is then calculated as

$$V_n = V_{n,max} - V_{av} . \quad (4.2)$$

The intensity of the second sound signal V_{2s} is determined by Eq. (4.3):

$$V_{2s} = V_{2s,max} - V_{av} , \quad (4.3)$$

where $V_{2s,max}$ is the voltage recorded on the peak of the signal.

The calculation for the time of flight (TOF, τ) of second sound is given after Eq. (4.4):

$$\tau = t_{2s} - t_0 , \quad (4.4)$$

where t_0 represents the time when the heater was turned on and t_{2s} when the measured voltage on the sensor increased significantly due to the detected temperature wave.

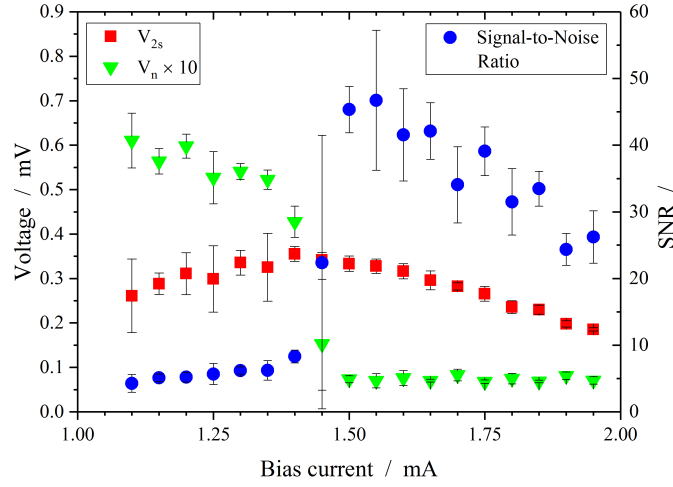


Figure 4.6: Signal-to-Noise-Ratio SNR, measured second sound signal V_{2s} and measured background noise V_n amplified by a factor 10 plotted vs. the bias current I of a Au20Sn225N1 at 1.8 K. The noise of the measurement decreases significantly when the sensor is biased with current higher than that of its maximum sensitivity (1.4 mA) leading to an increase of the SNR.

4.2.1 Signal-to-noise ratio

Based on the evaluation explained in the previous section, the signal-to-noise-ratio (SNR, ψ) can be calculated by Eq. (4.5)

$$\psi = \frac{V_{2s}}{V_n}. \quad (4.5)$$

The signal-to-noise-ratio represents the quality of the signal. It has already been reported in section 4.1.1, that the background noise can impede the second sound detection with TES. To study this, second sound pulses experiments were conducted under identical conditions (bath temperature, heater voltage, pair heater-sensor) except for the TES bias current. In Fig. 4.6, the measured SNR, detected second sound intensity and noise level resulting from this series of measurements are plotted as a function of the bias current for a TES type Au20Sn225N1 at $T = 1.8$ K and at a distance of 48.6 mm of an SMD heater pulsed with 30 V.

As expected, the intensity of the signal follows the same trend as the measured sensitivity curve (see 4.2(B2)) with respect to bias current. V_{2s} and sensitivity are proportional.

The maximum signal with 0.355 mV is reported for a bias current of 1.40 mA, which is in good agreement with the bias current for the highest sensitivity, which lies at 1.50 mA. For bias currents below 1.5 mA, the measured noise is in the order of 50 μ V to 60 μ V. Hence, the SNR is below a value of 10, despite of the expected high sensitivity. Only when sensors are biased with current higher than 1.5 mA, the background noise drops to approximately 10 μ V, resulting in a much bigger SNR of roughly 50. When the current is even increased further, the SNR decreases due to the decline of the signal intensity, while the noise level stays constant and low.

The exact reason for the high noise of the TES for bias currents below the one corresponding to the maximum sensitivity is unknown. A possible explanation could be interaction between the current source and the TES due to its high rise of voltage with current in this range leading to electrothermal feedback. Since the control of the current source is active, it could be possible that perturbations due to instabilities can be amplified non linearly by the control loop of the current source. It should also be mentioned that this effect was especially pronounced for TES with very high and narrow sensitivity curves while sensors with a moderate sensitivity on a large current range are more robust in what regards noise.

Based on these results, a biasing strategy for the TES can be developed. The TES has to be biased slightly above the current where the sensor measures the highest sensitivity to minimise the background noise but still obtain a sufficient sensitivity. It has also to be mentioned that even with this strategy, sometimes a big background noise ($> 100 \mu\text{V}$) is observed, which is visualised by the large error bars.

4.2.2 Validation of second sound

Although twelve TES were mounted on the support, only the three mounted Au₂₀Sn₂₂₅N₁ were used to measure the time of flight at $T = 1.8 \text{ K}$. The thermal sensitivity of sensors with other compositions did not provide a suitable measurement. The measured velocity of second sound $u_{2,m}$ was calculated based on the TOF τ obtained from Eq. (4.4) by

$$u_{2,m} = \frac{d_m}{\tau}, \quad (4.6)$$

where d_m is the actual known distance between the sensor and the heater.

In Fig. 4.7 the distance between the heaters and each of the three sensors is plotted as a function of the measured TOF. The linear trend is remarkable and the results coincide for all three sensors. The linear fit shown in the figure is given by

$$d = (19.5 \pm 0.5) \text{ m s}^{-1} \times \tau + (0.4 \pm 0.6) \text{ mm}.$$

The slope of the fit reveals a second sound velocity of $(19.5 \pm 0.5) \text{ m s}^{-1}$ at 1.8 K , which is in 2 % agreement with the literature value 19.9 m s^{-1} [43]. The influence of the amplitude dependence of second sound is considered to be negligible, since the distance between heater and sensors is large. The small deviation can be explained by the uncertainty of the measured distances with a calliper and a combination of the limited time resolution of the DAQ module and the analysing algorithm (see Section 3.7). Since additionally the literature value lies within the confidence interval (95.45 %), it can be said that second sound was successfully detected, which validates the measurement technique and the computational analyses.

4.2.3 Distance measurements and trilateration

Evaluation of distance measurement

Instead of using the TOF data to calculate the speed of second sound, the measured TOF was used for localising the second sound emitter, pretending that we ignored its localisation

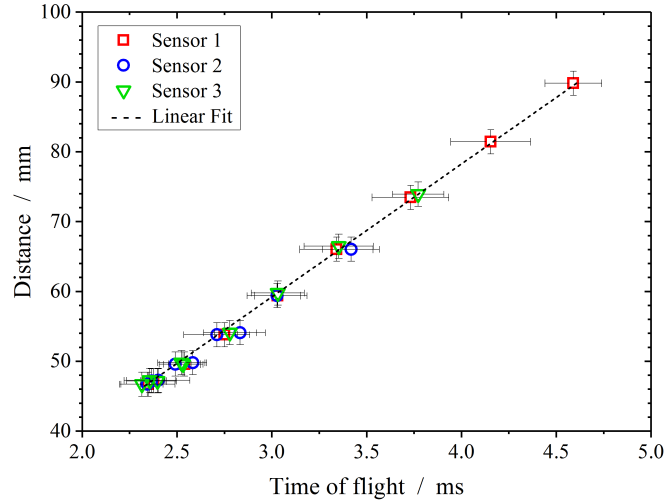


Figure 4.7: The distance between heater and sensors measured with a calliper plotted over the time of flight of the detected second sound by TES at a bath temperature of 1.8 K. The slope of the linear fit represents the measured velocity of second sound. With a value of 19.5 m s^{-1} , it is in good agreement with the literature value of 19.9 m s^{-1} [43].

and assuming that the speed of second sound is that which appears in the literature. With literature values of the velocity of second sound u_2 [43], the distance between the sensor and the corresponding heater is calculated from Eq. (4.7)

$$d_{2s} = u_2(T) \times \tau. \quad (4.7)$$

In Fig. 4.8, this calculated distance is shown for one TES at 1.8 K and compared to the value obtained from the calliper measurements. Sensor 3 was chosen, because it covers the longest distance range to the heater due to its position in the mounted setup. The two measurements of distance by the two methods are in excellent agreement, all pairs present a discrepancy of less than 2 mm. The TES is therefore capable to provide more the spatial resolution than an OST [90], even without additional amplification and DAQ systems with ultra high sampling rate ($> 100 \text{ kHz}$).

This outstanding result can be explained by the tiny size of the sensing region which is below 1 mm^2 . The exact position where the sensor detects the second sound is given more precisely for a TES than for an OST, which uses a foil with an area of 3 cm^2 .

Trilateration

Although the results of the previous section already indicate the capability of a TES to localise the second sound emitter, the results of different sensors were used to finally track the exact position of the heaters by trilateration. Since the focus of this thesis lies of the development of the sensors and second sound studies, a simple trilateration algorithm (see Apendix C.2) is chosen to illustrate the localisation of the second sound emitter. In order to be able to use more TES, the experiment was conducted at 2 K. Hence, additional to the three AuSn225N1 sensors, also two TES with a composition of 20 nm Au and 250 nm Sn

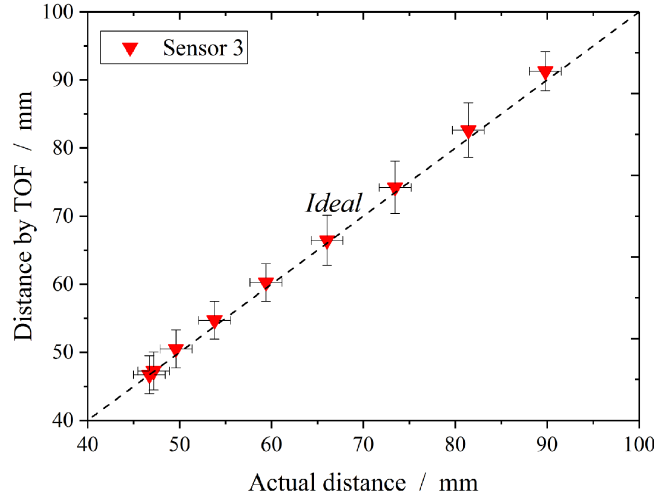


Figure 4.8: The calculated distance between the second sound emitter and the TES based on the second sound detection at $T = 1.8$ K plotted versus the actual distance, measured with a calliper. The dashed line displays the ideal case, where both values coincide. The used TES localises all of the nine emitters within 2 mm precision.

were used. Due to the suboptimal sensitivity of these sensors, a computational filter was applied to prevent wrong measurements due to noisy signals. Only when the calculated SNR of the measurement was above a value of 10, the measured TOF was used to locate the heater. In Fig. 4.9 a 2D map with all located spots is shown. Each point was determined by a trilateration calculation with a combination of three different sensors. The position of the 5 sensors used as well as the true location of the heaters are also drawn on the map.

The value of the Z-axis is not displayed, since the origin of second sound, no matter if a defect on a cavity or a heater, is always fixed to a surface whose position is known. With the resulting values in X- and Y-direction, the spot originating the wave can therefore be localised by extending the line perpendicular to the surface of the tested object. However, it should be mentioned that in most cases a value of ± 46 mm in Z-direction was calculated, which is in good agreement with the distance between the heater plate and the sensor support of 48.7 mm.

In Fig. 4.9, most of the points lie within the rectangle which illustrates the heater, meaning that the source was located correctly. However, although the TES measure the distances with high precision, there are still some points with an error of several millimetres, especially for Heater 5. Table 4.4 summaries the statistical result of the trilateration experiment.

In more than 92 % of the cases, the heater was localised within a precision of 2 mm. The discrepancy for the remaining cases can be explained by the performance limits of the trilateration algorithm used. Since it does not consider the impact of the uncertainty of the measured distance by the TES and only uses the results of three sensors instead of more (so-called multilateration [91]), already small uncertainties in the measured distance can lead to large deviations, especially when two of the three sensors are close to each other. There are advanced algorithms for quench localisation with OST [90], which can also be

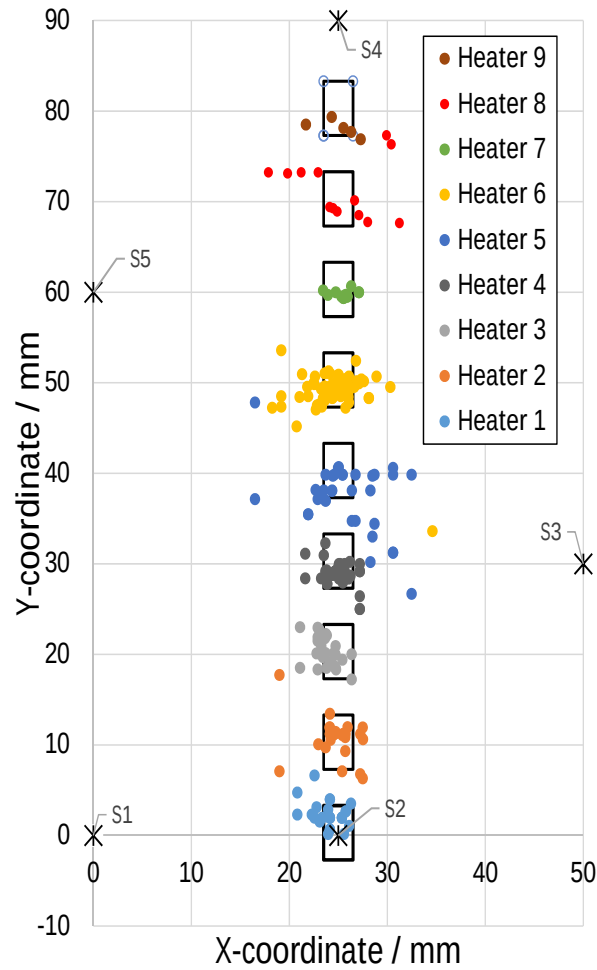


Figure 4.9: 2D map showing the spots where the TES network localised the corresponding second sound source. The position of the 5 TES used as well as the actual position of each heater is displayed, too.

Table 4.4: Statistical summary of the trilateration experiment at $T = 2\text{K}$, sorted by deviation between the localised spot and the actual position of the heater. The total amount of tracked points is $N = 264$.

	Deviation between localised and actual heater position			
	0 mm	< 2 mm	< 4 mm	> 4 mm
Absolute amount	219	24	10	11
Relative amount	82.95 %	9.09 %	3.79 %	4.12 %

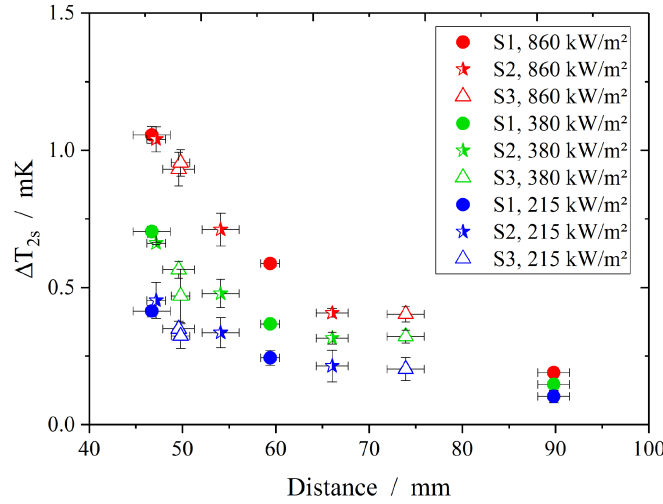


Figure 4.10: The measured temperature rise ΔT_{2s} with three different TES for three different heat fluxes over a distance range of approximately 46 mm to 89 mm. A clear decay of the intensity of the wave with increasing distance can be observed.

applied to TOF data of a TES network.

4.2.4 Influence of heat pulse intensity

With the thermal sensitivity data obtained from Section 4.1.1, it is possible to calculate the temperature change ΔT_{2s} on the TES due to second sound

$$\Delta T_{2s} = \frac{V_{2s}}{\left(\frac{\partial V}{\partial T}\right)}. \quad (4.8)$$

In Fig. 4.10 the measured temperature rises with 3 TES type Au20Sn225N1 at $T = 1.8$ K for three different heat fluxes are shown. The fact that all of three sensors follow the same trend for a given heat flux demonstrates that calculating the thermal sensitivity from steady state measurements is a valid method to translate voltage to temperature variations during dynamic measurements.

As expected, the highest heat flux of 860 kW/m^2 causes close to the emitter the biggest temperature raise of 1.1 mK. The trend that lower heat flux causes a less intense second sound wave can be explained from theoretical study of second sound waves [92]:

$$\Delta T = \frac{\dot{q}}{\rho_b u_{2,0} c_p}. \quad (4.9)$$

However, this equation only applies *fully* for a planar second sound wave. For the case in this thesis, due to the spherical propagation of second sound in 3D, other factors have to be taken into account (see Section 2.3.2).

It can be observed that for each heat flux, the intensity of the temperature wave falls with increasing travelling distance. For a distance of 90 mm, the measured temperature change for the highest and the lowest heat flux only differs by $87 \mu\text{K}$. After Atkins, the decay of

the amplitude of the second sound wave should follow a trend of r^{-1} (see Eq. (2.20)). By fitting the curves for the three heat fluxes with a function

$$y = Kx^{-n}, \quad (4.10)$$

the resulting exponent n is 2.59 ± 0.28 , 2.28 ± 0.28 and 2.63 ± 0.45 for a heat flux of 860 kW/m^2 , 380 kW/m^2 and 215 kW/m^2 , respectively.

A reason for this much faster than expected decay of temperature could be the fact, that in the testing setup used distance and angle between second sound source are linked. While the sensor is really in front of the heater for the shortest distance measured, the angle grows for longer distances. This could be explained by the fact that the intensity is not uniform on the spherical propagation front, but depends on the angle between the line of sight emitter-sensor and the perpendicular to the emitter's surface even for constant distance (see Section 2.3.1).

4.3 Angular dependence of second sound propagation

From what was discussed at the end of the previous section, it was decided to do second sound measurements in which the distance to the heater and the angle between the line of sight and the heater's normal were independent. This would allow to discriminate to which extent the strong dependence of intensity on distance reported in the previous section can be exclusively attributed to distance.

To do this the setup described in Section 3.6.1 was used. Four pairs of OST and TES (type Au20Sn225N1) were used on the described supports. Two experimental campaigns were followed: one in which all the sensor pairs lay on a single circle of roughly 85 mm away from the heater on the stand pipe; another in which each of the four sensor pairs is placed at a different distance from the stand pipe.

As a result, measurements were performed by varying many parameters: the distance from the sensor to the emitter, the propagation angle, the size of the heater and the type of sensor.

4.3.1 Effect of the propagation angle at different distances

In Fig. 4.11, the results of the intensity measurement for constant (left) and different (right) distances, at 1.8 K are shown. A large SMD heater ($8 \text{ mm} \times 7 \text{ mm}$) was used to produce a heat flux of 366 kW/m^2 . Both figures show clearly a decay of the measured second sound intensity as the angle increases. The maximum intensity is always measured for an angle of 0° , when the TES is facing the heater. A flat region from -10° to 10° can be observed. Around 20° the slope of intensity as a function of angle is maximum, beyond 20° the decay is similar to an exponential one, with decreasing slope as the angle increases. The signal is still significant at 90° , and not tending to 0.

The graph in which all TES had the same distance of 84.5 mm to the heater, the maximum intensity measured is between $(1.25 \pm 0.06) \text{ mK}$ and $(1.38 \pm 0.08) \text{ mK}$. As expected, the

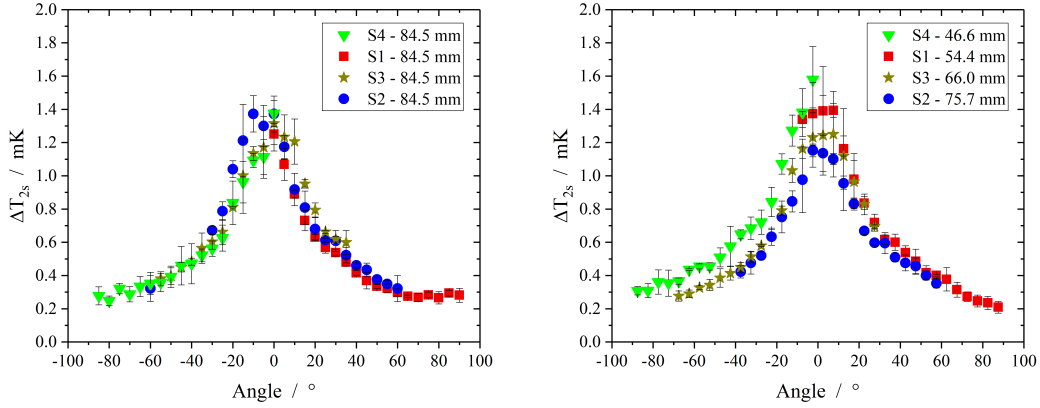


Figure 4.11: Measured temperature change with TES over different angles for constant (left) and varying (right) distances at 1.8 K. A large SMD heater ($8\text{ mm} \times 7\text{ mm}$) was used to produce a heat flux of 366 kW/m^2 . A decay of the intensity with increasing angle in both cases can be observed. The nomenclature S1 to S4 represents each of the four TES which were used in both experiments.

values for the different sensors are mostly overlapping, small deviations for angles lower than 5° can be explained with the uncertainty of the angle measurement (the x-error bars are not plotted for better visibility, the values can be taken from Appendix D.1). For sharp angles of less than 5° , as shown in both graphs, a small error during the angle adjustment has a more intense influence than for larger angles.

In the experiment where the distances of each sensor to the heater are different, the maximum temperature changes measured for the four TES are $(1.39 \pm 0.04)\text{ mK}$, $1.15 \pm 0.04\text{ mK}$, $(1.25 \pm 0.26)\text{ mK}$ and $(1.58 \pm 0.20)\text{ mK}$, respectively. The nomenclature S1 to S4 describes each of the four TES which were used in both experiments. Since all the TES are placed at a distance closer as 84.5 mm to the heater, it was expected to measure higher temperature fluctuations with all four of them. However, only S4, which is the sensor closest to the heater with a distance of 46.6 mm, lies clearly above the values measured in the first experiment.

Based on the data from Fig. 4.11, the decay of temperature with increasing distance is plotted for four angles in Fig. 4.12. It has to be mentioned that due to the fact that the heater surface is not exactly at the center of the circle, the distance between heater and sensors change between 1 mm and 7 mm when changing the angle. When the heater is really in front of the sensor, the steepest slope with an exponent $n = (0.640 \pm 0.025)\text{ mK mm}^{-1}$ is observed. For angles of 20° , 40° and 60° , n results to $(0.522 \pm 0.040)\text{ mK mm}^{-1}$, $(0.541 \pm 0.078)\text{ mK mm}^{-1}$ and $(0.628 \pm 0.090)\text{ mK mm}^{-1}$, respectively.

The comparison of the measured values with the TES in this work compared to those published by Zhang [52]—obtained with glass fibre substrate TES probes—are roughly 15 times lower. Although the power and surface of the heater are different, as well as the bath temperature, it is not a probable explanation for such a big discrepancy. It is more plausible that the Kapitza resistance [93] between the helium and the thin film and the latter's high thermal coupling to the glass substrate—highly diffusive and quite thick,

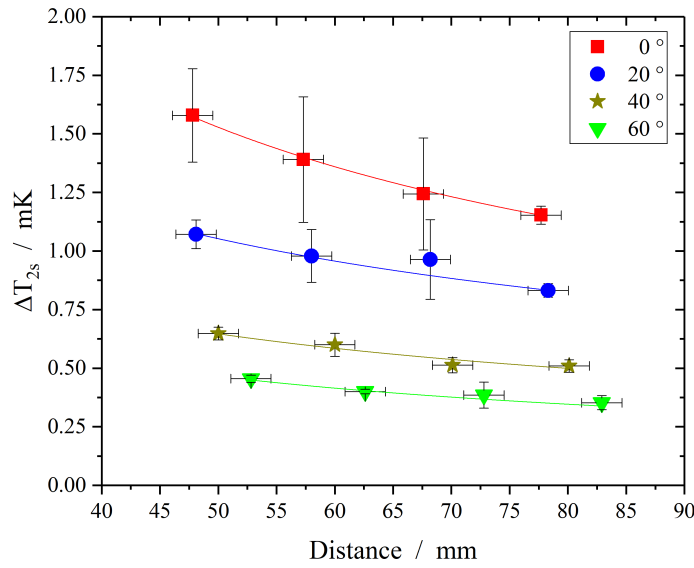


Figure 4.12: Measured temperature change with 4 TES, each located at a different distance to the heater. The results are plotted for four different fixed angles. With increasing angle, the decay flattens. A fit $y = Kx^{-n}$ for each angle is shown in the corresponding colour as well.

0.5 mm—result in a not fast enough thermal response with respect to the duration of the temperature fluctuation peak. This needs to be further investigated by correctly modelling the propagation of second sound in 3D from a planar heater as well as the thermal response of the sensor to fluid temperature fluctuations.

However, the scope of this thesis is to show empirically how the *detected* intensity behaves as function of the distance. To conclude, it can be shown that the decay of the peak intensity of the detected second sound wave with increasing distance is much more modest than in the parallel plate experiments discussed in Section 4.2.4 (exponent $n > 2$) and most of the decrease is attributed to the increasing angles for longer distances in this setup.

4.3.1.1 Influence of the heater size

Fig. 4.13 shows the result when instead of a large 8 mm × 7 mm heater a small heater with the size of 6 mm × 3 mm was used.

It is clearly visible that for the small heater the TES detected much lower temperature changes, even if the power applied on the heater is the same (within 10 %) and the heat flux is increased in more than a factor 2. For an angle of 0°, the maximum temperature fluctuation measured is (0.21 ± 0.02) mK, whereas it is more than 1.2 K for the big heater. The decay of the temperature with increasing angle is also much smaller. With a value of (0.13 ± 0.01) mK, the difference is only (0.08 ± 0.03) mK at an angle of 80°.

At first sight, this result is contrary to the one expected. Due to the higher heat flux, the second sound peak intensity should be higher than for the big heater. Hence, it was expected that the TES measured a higher temperature when being perpendicularly in front of the

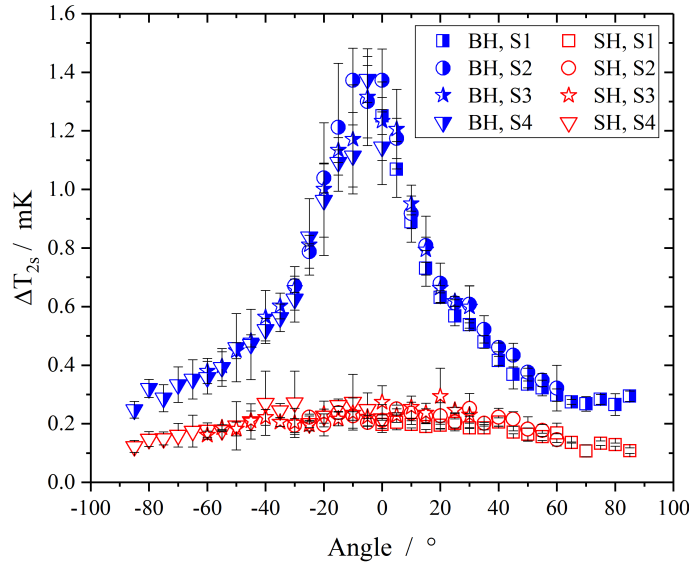


Figure 4.13: Comparison between the temperature change measured with TES for a big heater (BH, 8 mm × 7 mm) and a small heater (SH, 6 mm × 3 mm) at 1.8 K. The heat flux produced by the small heater is 689.8 kW/m², whereas the big heater reaches a value of 366 kW/m². Contrary to the results of the big heater, the measured temperatures for the small heater are a factor six lower and decay slower with increasing angle.

heater. For greater angles, the signal should decrease fast, since the planar part of the wave is smaller than that of the big heater. Zhang *et al.* showed this effect with their simulations as well (see Appendix: E.3). A reason for the unexpected low temperatures could be that this high intense, planar part of the wave is so small, that due to misalignment, it does not “hit” the sensor. The sensor only measures the temperature of the residual, radial part of the wave, in which the temperature gradient is low.

4.3.1.2 Comparison between TES and OST

The amplitude of the signal for an OST measurement is obtained by calculating the slope of the peak m_{2s} with Eq. (4.11)

$$m_{2s} = \frac{V_{2s,min} - (V_{av} + V_n)}{t_{2s,min} - t_{2s}}, \quad (4.11)$$

where $t_{2s,min}$ is the time of the recorded minimum of the peak. This method was chosen, because the time resolution of the Oscilloscope (0.004 ms) is much higher than its limited voltage resolution (4 mV, see Appendix D.1).

It is known that the intensity response of an OST is highly dependent on the fixation of the membrane when mounting it. This is difficultly reproducible. As the voltage delivered by an OST cannot be, by calibration, directly linked to a controlled temperature fluctuation or relative inter-component velocity, the measured intensities needed to be normalised in order to be able to do a comparison between different OST’s and with the TES. It was chosen to do this normalisation by dividing any measured intensity ξ by a given OST with

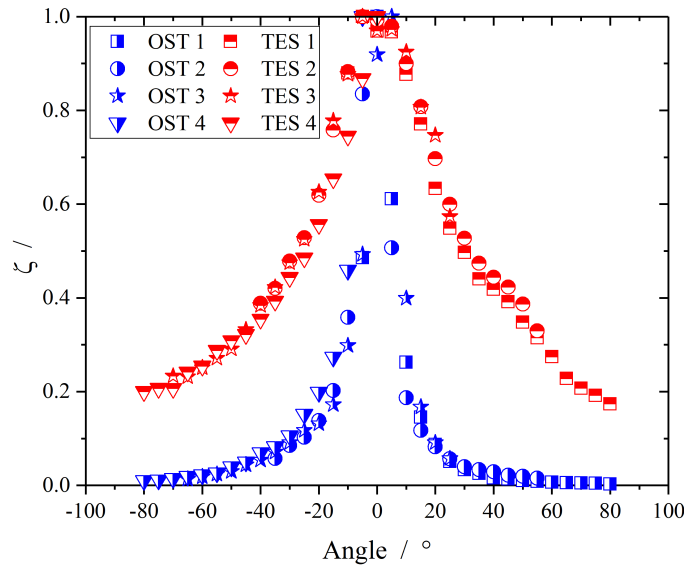


Figure 4.14: Comparison of the measured signal amplitude for OST and TES at 1.8 K. A large SMD heater ($8\text{ mm} \times 7\text{ mm}$) was used to produce a heat flux of 208 kW/m^2 . For each sensor, the measured intensity is normalised by the maximum signal measured. While both sensors measure the maximum amplitude when being perpendicular to the heater, the signal measured by the OST decays much faster than that of the TES.

respect to its own intensity in the case of zero propagation angle, for the same parameters (heat flux on heater, bath temperature, distance). Equivalently, it was considered that the highest intensity measured at zero angle ξ_{max} gives the nominal sensitivity ζ of the OST to a given event ξ :

$$\zeta = \frac{\xi}{\xi_{max}} . \quad (4.12)$$

In case of the TES, the amplitude ξ represents ΔT_{2s} , whereas for the OST it is the slope of the first peak (see Eq. (4.11)). In Fig. 4.14 the results for a measurement with OST and TES at 1.8 K are shown. A large SMD heater ($8\text{ mm} \times 7\text{ mm}$) was used to produce a heat flux of 208 kW/m^2 . As expected, both sensors measure the maximum signal when facing the heater at zero propagation angle. However, the signal of the OST decays much faster than that of the TES as the angle is increased. For an angle around 60° , ζ shows a value of approximately 0.02. This means that the measured maximum signal at 0° is around 50 times higher than the signal at 60° . The fact that the decay is slightly faster for positive than for negative angles could be caused by the positioning of the heater. The heaters had to be glued on the support. It is therefore possible, that a non-uniform distribution resulted in a small tilting of the heater.

In the case of the TES, for an angle of 80° , the measured signal still has a relative intensity of 0.2, whereas for the OST ζ is practically zero.

The difference in shape of the angular dependance of the TES with respect to the OST can be explained taking into account the nature of the detection process of each sensor.

The TES are thermometers with a finite response time constant. When the signals are too short in duration, a significant fraction of their intensity is filtered. This effect is expected to be higher at small angles where the pulse duration is expected to be extremely short, compared to that at high angles.

The OST measure the inter-component velocity. As such, if this vector is not perpendicular to the OST membrane, the detected intensity will be lower. It is to expect that for a planar heater, the relative velocity vector will not be perfectly radial with respect to the heater, especially at big angle [52], with which the excitation of the membrane decays fast with the angle.

Both effects tend to make the OST angular response more peaked than the one of the TES.

5. Conclusion

5.1 Summary of results

In this study new prototypes of transition edge sensors (TES) were successfully produced and validated as second sound detectors. By investigating TES of different composition with SEM and EDS, important correlations between the microstructure and the superconducting behaviour of the TES could be observed. The higher critical current and steeper transition for TES with increased amount of tin could be traced back to the increasing size of grain-like, tin-rich features and the reduction of the distance between them within the thin film.

Additionally, it was successfully demonstrated that the superconductivity of the TESs is enhanced by annealing the sensors at 120 °C for 10 minutes. EDS analyses of the thin film revealed that the distribution of the two components gold and tin became uniform. All this information can be used to adjust the manufacturing process of the TES based on the obtained microstructure to receive TES with the desired properties. Moreover, sensors with insufficient superconductivity could be corrected by heat treatment.

Sensors with a tin thickness between 200 nm and 250 nm on 20 nm showed a sufficient thermal sensitivity for second sound detection above 1.6 K. Especially TES with a composition of 20 nm gold and 225 nm tin showed excellent performance in the typical SRF cavity testing temperature range of 1.8 K to 2.0 K. The time of flight of heat pulses emitted by SMD heaters was measured with several TES at a known distance and the obtained second sound velocity of $(19.5 \pm 0.5) \text{ m s}^{-1}$ at 1.8 K is in good agreement with the literature data of Donnelly [43].

Based on this literature value of propagation speed of second sound, the time of flight of the temperature wave was used to calculate the distance between TES and SMD heaters at 1.8 K. Comparing the results to the actual values showed that all distances could be estimated within 2 mm precision.

When repeating the experiment at 2.0 K, a trilateration algorithm was used to localise the exact position of the heaters. Despite of the simplicity of the algorithm, 92.04 % of the heaters were located within a spot 2 mm proving the capability of TES as quench localisation devices. This is even more outstanding taking into account that only a DAQ system with moderate time resolution was used and no post processing of the signal like amplifying or noise filtering was needed.

In a second set-up, new insights in three-dimensional propagation of second sound could be achieved. The temperature of the second sound wave for different angles was successfully measured with TES optimally biased after a self-developed strategy. An influence of the angle with respect to the second sound emitter as well as the size of the emitter on the measured amplitude of the wave could be recognised. It was demonstrated that the decay of the amplitude is not only dependent on the distance but also, and in a very pronounced

fashion, on the angle between the line of sight of the sensor and heater. Finally, a comparison between TES and OST measurements proved the different kind of second sound detection for the two sensor types, revealing that an OST, contrary to a TES, can not simply be calibrated to a temperature measurement device.

5.2 Outlook

To improve the production process of the TES in the future, more straightforward metallisation techniques can be used to increase manufacturing speed and reproducibility. TES consisting of a thin Au-Sn film deposited by sputtering have already shown promising results as second sound detectors in first tests at CERN.

Although the quench localisation with TES has already been successfully proven, the accuracy can still be improved by upgrading the time resolution of the DAQ equipment. Furthermore, a filter and amplification system such as the one of the OST can be added to minimise the level of background noise during the measurement.

The propagation of second sound waves in different directions should still be investigated more thoroughly. Especially the formulation of quantitative correlations between distance and angle dependence are important with regard to the quenching behaviour of SRF cavities. Additionally, the experiments could be repeated with another type of highly sensitive thermometer, e.g. CernoxTM *bare chip* CX-1030 [94], to check the temperature measurement of the TES. However, although CernoxTM sensors can detect second sound, they are not suitable for SRF cavity testing due to their fragility and should only be used for cross checking against the TES.

To improve the time response of the TES, a new design can be developed decreasing the amount of the glass substrate below the thin film. Instead of the borosilicate wafer, glass fibre probes could be used.

To conclude, it is important to mention that TES are already installed in the SM18 testing facility of CERN to study the usage of TES as a quench localisation system in a real SRF cavity performance test. First results are expected soon.

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Appendix

A Material properties

A.1 Specific heat of liquid helium

Fig. A.1 shows the characteristic shape of the plot of specific heat C as a function of temperature near the transition from He-I to He-II.

A.2 Au-Sn phasediagram

In Fig. A.2 the phase diagram of Au-Sn is displayed [95].

A.3 Temperature scale of helium II at saturated vapour pressure

The temperature of helium II above 1.25 K can be expressed as a function of its vapour pressure, which has been adopted as “The international Temperature Scale of 1990” (ITS 90) [102]

$$T/\text{K} = A_0 + \sum_{i=1}^9 A_i \left[\frac{(\ln(p/\text{Pa}) - B)}{C} \right]^i \quad \text{for } 1.25 \text{ K} < T < T_\lambda \quad (5.1)$$

where A_i , B and C are coefficients listed in Table A.1. In this thesis, (5.1) was always used to calculate the bath temperature T_0 .

B Physical measuring methods

B.1 Profilometer

A profilometer is a tool which measures the profile of a surface to determine its roughness. There are two big groups of profilometers: the optical profilometry and the contact profilometry. The most famous contact method is the stylus profilometer, also called mechanical profilometer. A tiny sharp diamant needle moves along the probe touching its surface and recording the change of its vertical position due to the roughness of the surface. The measurement accuracy of a stylus profilometer is in the order of a few nanometres. [100, 101]

Table A.1: Empirical parameters of the ITS 90, valid for $1.25 \text{ K} < T < T_\lambda$ [102]

Coefficient	Value	Coefficient	Value
A_0	1.392408	A_6	-0.017976
A_1	0.527153	A_7	0.005409
A_2	0.166756	A_8	0.013259
A_3	0.050988	A_9	0
A_4	0.026514	B	5.6
A_5	0.001975	C	2.9

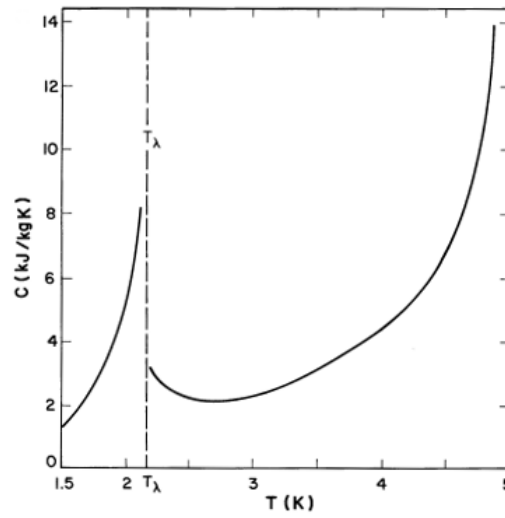


Figure A.1: Specific heat of liquid helium as a function of temperature at saturated vapour pressure. The characteristic shape of the Greek letter lambda is clearly visible [25].

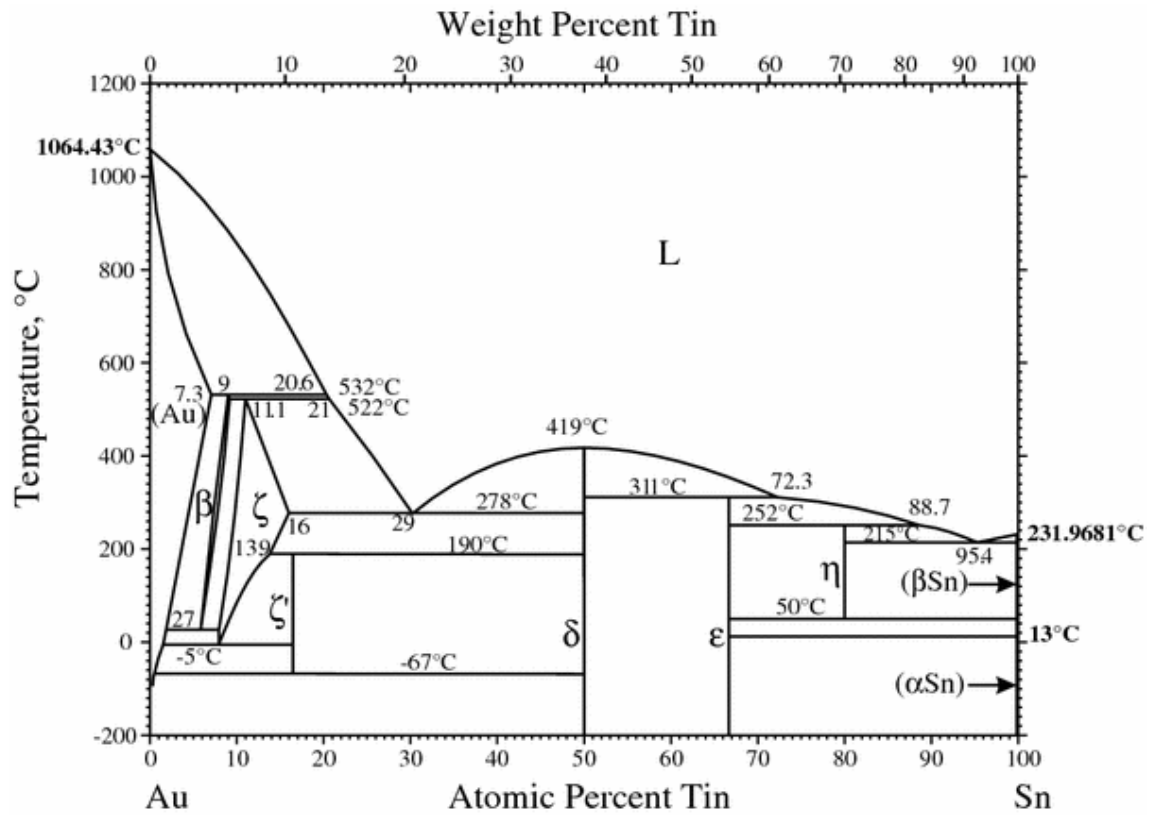


Figure A.2: Phase diagram of Au-Sn after [95]. The three important alloys for Au-Sn TES are δ : AuSn, ϵ : AuSn₂ and η : AuSn₄.

B.2 Scanning electron microscopy

The scanning electron microscopes (SEM) is nowadays an established tool for precise morphologic analyses. It outperforms a normal light microscope in aspects of resolution at high magnification and rendering of the topography.

A SEM normally consists of a microscope column, where the probe will be scanned, a computer for controlling the microscope and rendering the image and optional equipment for further analyses.

In the microscope column, an electron gun with a filament produces thermoelectrons. The electrons are then accelerated through the column by a high voltage between an anode and the filament. The resulting electron beam interacts with the atoms of the specimen producing various characteristic signals which can be detected. In most SEMs, the surface imaging is done by detecting secondary electrons, which have been mainly ejected from the surface of the sample by the electron beam. While the beam is scanning the surface of the specimen in a raster pattern systematically, a detector counts the amount of the ejected electrons. Since the amount of the electrons depends on the topography of the sample, a software can display a greyscale image of the surface of the specimen based on the locally varying electron contrast. To inhibit diffraction of the secondary electrons on other molecules, the SEM usually operates under high vacuum. [96, 97]

B.3 Energy-dispersive X-ray spectroscopy

As mentioned in the section above, an SEM can be upgraded with several tools to gain additional information about the sample. A common example for one of these devices is the energy-dispersive X-ray spectroscopy (EDS). In combination with an SEM, the EDS allows an elemental analysis of the specimen.

The EDS working principle is based on the fact that every element has its unique atomic structure. By focusing an electron beam on the sample, electrons in the inner shell of the atoms are getting excited and finally ejected. This leaves a whole in the shell which will be filled from an electron of a higher-energy shell of the atom. The energy difference of this transition between the outer and the inner shell is then compensated by emitting an X-ray. Because the X-rays are characteristic for the energy difference of the shells and consequently also for the atomic structure, EDS is able to detect the presence of the elements in the sample. Moreover, by counting the amount of each characteristic X-ray, a quantitative composition analysis is possible. [96, 98, 99]

C Materials and methods - additional information

C.1 4-wire-measurement of TES

A schematical drawing of the 4-wire-measurement of the TES is shown in Fig. C.1. By separating the current and voltage electrodes, the lead resistance of the cables can be eliminated from the measurement.

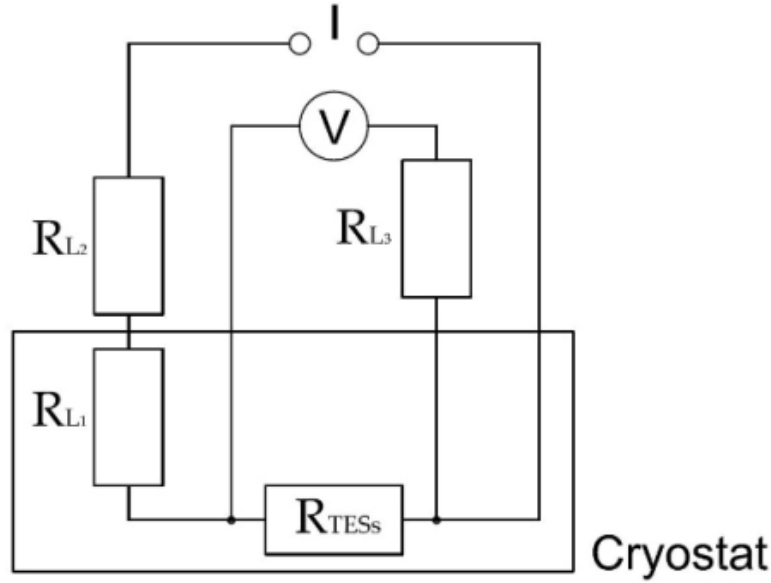


Figure C.1: Schematical graphic of the 4-wire-measurement circuit of the TES [6].

C.2 Trilateration algorithm

A simple trilateration algorithm [103] was used for to calculate the results in Section 4.2.3. The positions of three sensors $P1$, $P2$ and $P3$ are expressed in a transformed Cartesian coordinate system. r_1 , r_2 and r_3 represent the calculated distance to the heater by second sound detection for each sensor. A sphere is drawn around each sensor with the corresponding radius

$$\begin{aligned} r_1^2 &= x^2 + y^2 + z^2 \\ r_2^2 &= (x - k)^2 + y^2 + z^2 \\ r_3^2 &= (x - l)^2 + y - m^2 + z^2 \end{aligned} \tag{5.2}$$

where x , y and z are the coordinates of the intersection point of the three spheres which represents the located heater. After rearranging the equations, an expression for the coordinates results

$$\begin{aligned} x &= \frac{r_1^2 - r_2^2 + k^2}{2k} \\ y &= \frac{r_1^2 - r_3^2 - x^2 + (x - l)^2 + m^2}{2m} \\ z &= \sqrt{r_1^2 - x^2 - y^2}. \end{aligned} \tag{5.3}$$

To simplify the calculation, the positions have to be expressed as vectors from the origin where indicated. The unit vectors for the three directions of the transformed coordinate

system are then given as

$$\begin{aligned}\hat{e}_x &= \frac{P2 - P1}{\|P2 - P1\|} \\ \hat{e}_y &= \frac{P3 - P1 - k\hat{e}_x}{\|P3 - P1 - k\hat{e}_x\|} \\ \hat{e}_z &= \hat{e}_x \times \hat{e}_y\end{aligned}\tag{5.4}$$

Since all the sensors are glued to the same plate, the coordinate system was chosen in such a way that all sensors are placed on the x-y-plane. Furthermore, it was assumed that the z-coordinate of the heater is always positive. This assumption is valid since we know on which side of our sensor the object which should be localised actual is. l is the signed magnitude of the x component in the transformed coordinate system

$$l = \hat{e}_x (P3 - P1).\tag{5.5}$$

The signed magnitude of the y component in the transformed coordinate system results in

$$j = \hat{e}_y (P3 - P1).\tag{5.6}$$

Moreover, the distance between $P1$ and $P2$ is needed

$$k = \|P2 - P1\|.\tag{5.7}$$

With this formula (5.3) can be solved in the transformed coordinate system. The coordinates of the located heater $\vec{p}_{1,2}$ in the actual system are then given by

$$\vec{p}_{1,2} = P1 + x \hat{e}_x + y \hat{e}_y + z \hat{e}_z\tag{5.8}$$

C.3 OST amplifiers

In Fig. C.2 the circuit layout of the OST box to amplify the measured signal and reduce noise is displayed.

D Evaluation of measurement uncertainty

D.1 Overview of measurement uncertainties in this work

Measurement uncertainty of devices used

An overview of all devices used and their corresponding measurement uncertainties is given in Table D.1.

Estimated type B measurement uncertainties

The uncertainties of measurands estimated after a type B evaluation are listed in Table D.2.

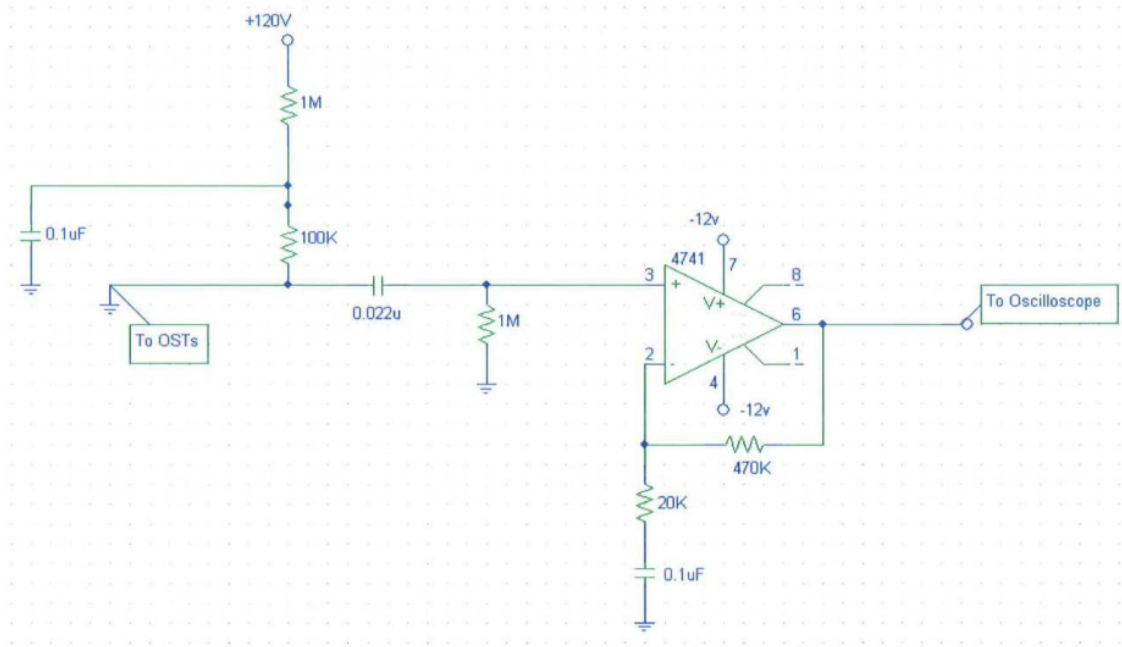


Figure C.2: Circuit layout of the OST amplifiers from [13].

Table D.1: Summary of the measurement uncertainties of the devices used. The given uncertainties already cover different influences, such as digitalisation, resolution and uncertainty claimed by the manufacturer.

Device	Parameter	Uncertainty	Unit
Pressure controller MKS Type 651C	p	$u_p = 0.0025 \cdot p + 1$	Pa
Current source Keithley TM 6220	I	$u_I = 0.0005 \cdot I + 0.001$	mA
Oscilloscope Tektronix DPO 3014	V	$u_{V,osc} = 4$	mV
Oscilloscope Tektronix DPO 3014	t	$u_{t,osc} = 0.004$	ms
DAQ modules NI 9251	V	$u_{V,ni} = 0.0092$	mV
DAQ modules NI 9251	t	$u_{t,ni} = 0.0488$	ms

Table D.2: List of the type B measurement uncertainty of additional measurands which have to be taken into account.

Description	Parameter	a	Uncertainty	Unit
Distance sensor - heater	d	3	$u_d = 1.73$	mm
Angle sensor - heater	α	1.5	$u_\alpha = 0.87$	°
Velocity second sound [43]	u_2	-	$u_{u_2} = 0.002 \cdot u_2$	m s ⁻¹

Table D.3: List of all parameters whose combined standard uncertainty has to be calculated on input uncertainties.

Description of parameter	Combined standard uncertainty
Temperature HeII bath	$u_{c,T} = f(u_p)$
Sensitivity of TES	$u_{c,Sens} = f(u_{c,T})$
Measured velocity of second sound	$u_{c,u_{2,m}} = f(u_\tau; u_d)$
Distance measured by TOF	$u_{c,d_{2s}} = f(u_\tau; u_{u_2})$
ΔT measured with TES	$u_{c,\Delta T} = f(u_{V_{2s}}; u_{c,Sens})$

List of combined standard measurement uncertainties

In Table D.3, the evaluated parameters of the present work whose uncertainty consist of different input uncertainties are listed. The detailed equations to calculate the uncertainties can be found in the next section. Theoretically, the measurement uncertainties of the current source, the oscilloscope and of the DAQ module had to be considered for the calculation of the combined standard uncertainties listed above as well. However, compared to the statistical measurement uncertainties u_τ and $u_{V_{2s}}$ they were negligibly small.

D.2 Detailed equations used to evaluate the uncertainty of measurement

The principle approach for the evaluation of uncertainty is given in Chapter 3.8. Based on the equation (3.6), the combined standard uncertainty was calculated for the corresponding measurand. A model equation has to be given, so that the sensitivity coefficients $\partial f / \partial x_i$ can be calculated.

The temperature of the helium bath is calculated after (5.1)

$$T/\text{K} = A_0 + \sum_{i=1}^9 A_i \left[\frac{(\ln(p/\text{Pa}) - B)}{C} \right]^i \quad \text{for } 1.25 \text{ K} < T < T_\lambda. \quad (5.1)$$

The uncertainty of the temperature measurement $u_{c,T}$ therefore only depends of the uncertainty of the pressure u_p

$$u_{c,T} = \sqrt{\left(\frac{A_1}{(p/\text{Pa}) C} + \sum_{i=2}^9 \frac{i A_i}{(p/\text{Pa}) C} \left[\frac{\ln(p/\text{Pa}) - B}{C} \right]^{i-1} \right)^2} u_p^2. \quad (5.9)$$

For the calculation of the velocity of second sound based on measurements, (2.10) was used

$$u_{2,m} = \frac{d_m}{\tau}. \quad (4.6)$$

The uncertainty $u_{c,u_{2,m}}$ is given by the uncertainties of the measured τ and the distance between heater and sensors d_m

$$u_{c,u_{2,m}} = \sqrt{\left(\frac{1}{\tau} \right)^2 u_d^2 + \left(-\frac{d_m}{(\tau)^2} \right)^2 u_\tau^2}. \quad (5.10)$$

The measured distance with the TES d_{2s} was calculated after (4.7).

$$d_{2s} = u_2 \times \tau . \quad (4.7)$$

The combined standard uncertainty $u_{c,d_{2s}}$ depends on u_τ and the uncertainty of the literature values of second sound u_{u_2}

$$u_{c,d_{2s}} = \sqrt{(u_2)^2 u_\tau^2 + (\tau)^2 u_{u_2}^2} . \quad (5.11)$$

The raise of the bath temperature caused by second sound measured by the TES is given as

$$\Delta T = \frac{V_{2s}}{\left(\frac{\partial V}{\partial T}\right)} . \quad (5.12)$$

The resulting combined uncertainty for the measured temperature results to

$$u_{c,\Delta T} = \sqrt{\left(\frac{1}{\frac{\partial V}{\partial T}}\right)^2 u_{V_{2s}}^2 + \left(-\frac{V_{2s}}{\left(\frac{\partial V}{\partial T}\right)^2}\right) u_{c,Sens}^2} . \quad (5.13)$$

The thermal sensitivity is calculated after Eq. (4.1)

$$\begin{aligned} \frac{\partial V}{\partial T}(I, T_j) &\cong \frac{T_j - T_{j-1}}{T_{j+1} - T_{j-1}} \frac{V(I, T_{j+1}) - V(I, T_j)}{T_{j+1} - T_{j-1}} \\ &+ \frac{T_{j+1} - T_j}{T_{j+1} - T_{j-1}} \frac{V(I, T_j) - V(I, T_{j-1})}{T_{j+1} - T_{j-1}} . \end{aligned} \quad (4.1)$$

It is important to mention that the error bars displayed in Section 4.3 were calculated with a k-factor of 1 instead of 2 for the thermal sensitivity. However, the error bars of the sensitivity data shown in Fig. 4.2 were calculated with a factor of $k = 2$.

Only the uncertainty of the temperature measurement is considered for the calculation of the uncertainty of the sensitivity

$$\begin{aligned} u_{c,Sens}^2 &= \sqrt{u_{c,T}^2 (A^2 + B^2 + C^2)} \quad \text{with} \\ A &= \left(\frac{V(I, T_{j+1}) - 2V(I, T_j) + V(I, T_{j-1}))}{(T_{j+1} - T_{j-1})^2} \right) \end{aligned} \quad (5.14)$$

$$\begin{aligned}
B &= \left(\frac{-2T_{j-1} [V(I, T_{j+1}) - 3V(I, T_j) + V(I, T_{j-1})]}{(T_{j+1} - T_{j-1})^3} \right) \\
&+ \left(\frac{2T_j [V(I, T_{j+1}) - 2V(I, T_j) + V(I, T_{j-1})]}{(T_{j+1} - T_{j-1})^3} \right) \\
&+ \left(\frac{[T_{j+1} (V(I, T_j) - V(I, T_{j-1}))]}{(T_{j+1} - T_{j-1})^3} \right) \\
C &= \left(\frac{2(T_j - T_{j-1})(V(I, T_{j+1}) - V(I, T_j))}{(T_{j+1} - T_{j-1})^3} \right) \\
&- \left(\frac{V(I, T_{j+1}) - V(I, T_j)}{(T_{j+1} - T_{j-1})^2} \right) \\
&+ \left(\frac{2(T_{j+1} - T_j)(V(I, T_j) - V(I, T_{j-1}))}{(T_{j+1} - T_{j-1})^3} \right)
\end{aligned}$$

E Results

E.1 Annealing

In Figs. E.1 and Fig. E.2 the recorded data of the two conducted annealing processes are shown. It can be seen that in both cases the actual sample temperature was 2 °C to 5 °C higher than desired.

E.2 Influence of heater size after Zhang

In Fig. 2.13 the simulated propagation of a second sound wave at 1.6 K for two heaters with different size (30×30 and 15×15) [52]. The black bar indicates the heater size.

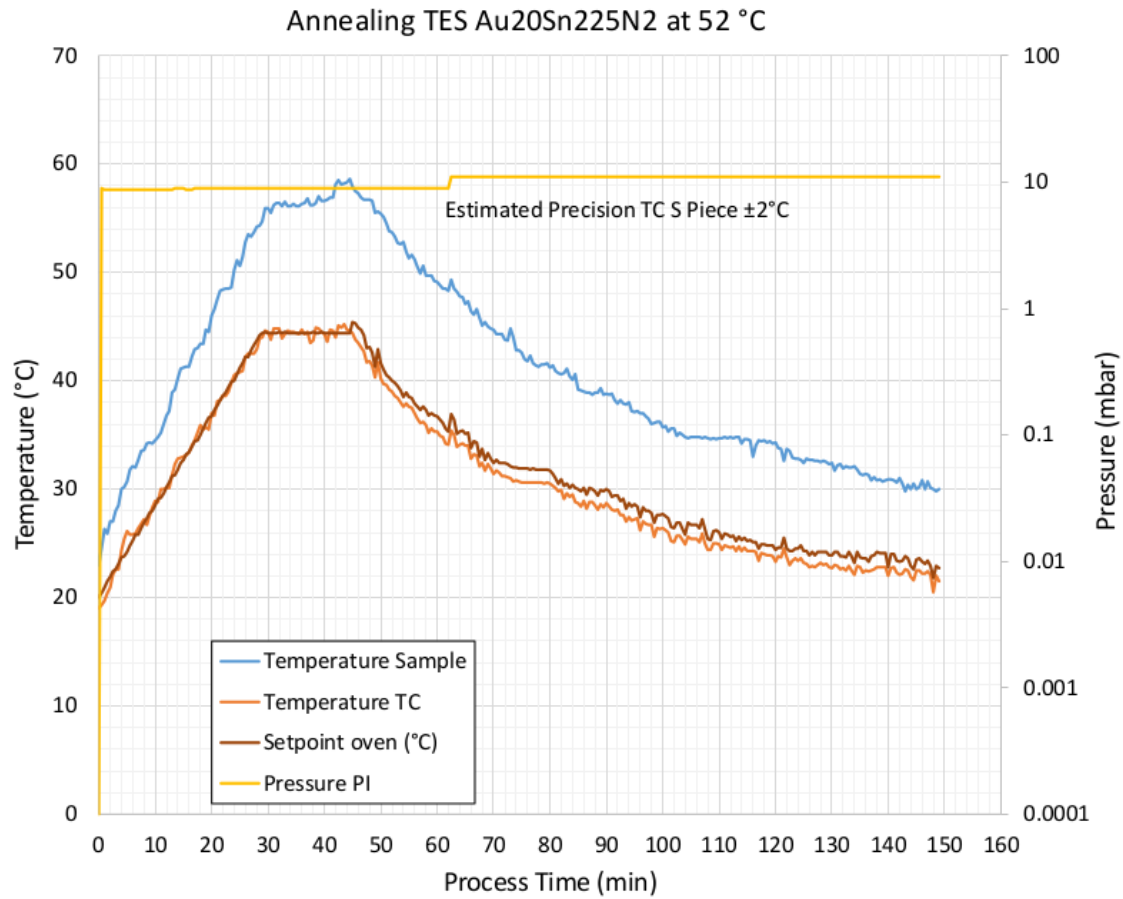


Figure E.1: Recorded data during the annealing process at 52 °C under argon atmosphere. Temperature TC is the temperature measured with a thermocouple to control the oven temperature, given by the setpoint. Since the VAS annealing oven is normally made for temperatures above 200 °C, the calibration of the temperature sensor is not fully correct. As a result, the actual temperature inside the oven can be higher than the temperature measured on the thermocouple.

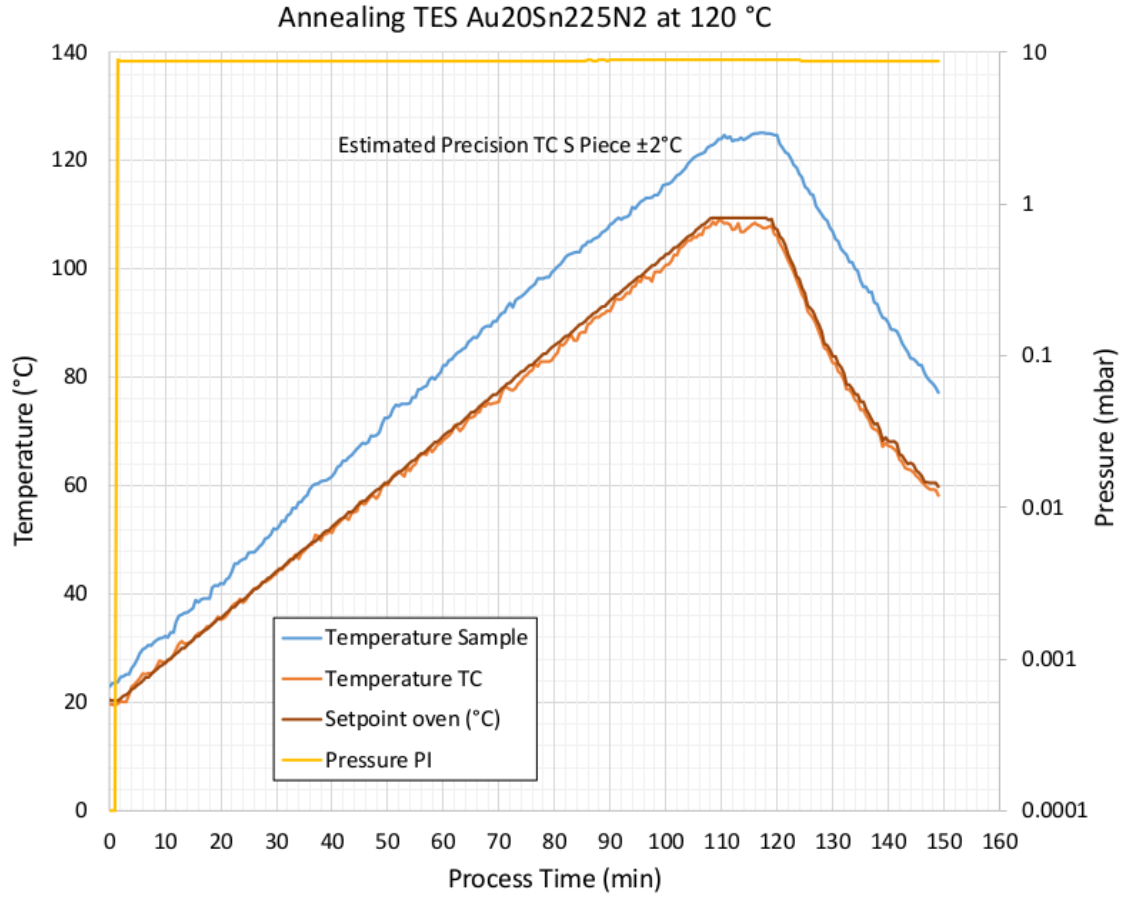


Figure E.2: Recorded data during the annealing process at 120 °C under argon atmosphere.

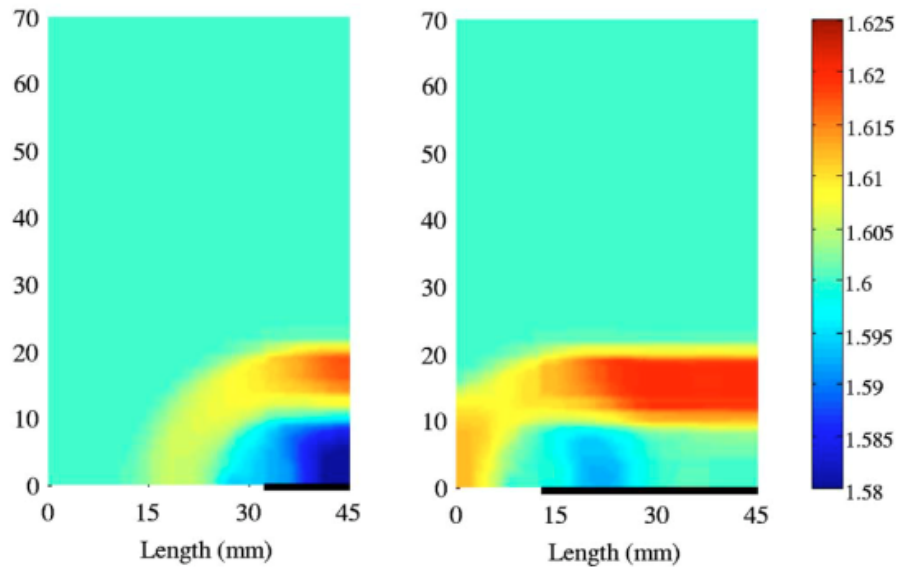


Figure E.3: 2D slides of a 3D pulsed second sound wave at 1.6 K for two different heater sizes [52]. The produced heat flux was 100 kW/m² and the heating duration was 0.5 ms in both cases. The black bar indicates the heater size.