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Josephson and noise scanning tunneling microscopy on conventional, unconventional and disordered superconductors

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Introduction

In this introductory chapter we aim at providing the reader with our motivation behind the work presented in this thesis on different families of superconductors investigated with Josephson and noise scanning tunneling microscopy. To this end, the relevant background concepts on superconductivity will be presented alongside with some historic allusions.

1.1. Superconductivity: it is all about pairing

Superconductivity is a phase of electronic matter in which the electrical resistivity in certain materials disappears upon their cool down below a characteristic critical temperature T_C. In 1911, Heike Kamerlingh Onnes (Nobel 1913) discovered superconductivity for the first time in mercury (Hg) ($T_C = 4.2$ K) [1]. At the time, his physics laboratory in Leiden University was the coldest place on earth since l908; the year in which Onnes and his co-workers liquefied helium[1] for the first time, opening the field of low temperature physics. [2] In the years that followed several, other low-temperature superconductors were discovered, including elemental lead (Pb) ($T_C = 7.2$ K) and niobium (Nb) ($T_C = 9.3$ K) as well as compounds like niobium nitride (NbN) ($T_C = 16$ K).

Subsequently, the works of the London brothers [2], Abrikosov [3], Ginzburg and Landau [4] were the first theoretical attempts to explain superconductivity. Despite the progress, it was not until the late 50s that the first microscopic theory of superconductivity was published.

Electron pairs in conventional superconductors

The first microscopic theory of conventional superconductivity appeared in 1957 by Bardeen Cooper and Schrieffer (BCS) [5]. For their pionnering work BCS received the Nobel prize in physics in 1972. BCS theory is a combination of the mean field approximation and the quasiparticle approach of Landau that describes the electronic many body interactions in a superconductor. In the work by BCS, it is shown that electrons attract each other, forming pairs, under the interaction via lattice vibrations; phonon exchange. A step further is taken to find that in the presence of an attractive interaction between electrons, the ground state of a superconductor below T_C is described as a coherent condensate of electron pairs, called *Cooper pairs*; in essence a *superfluid*. Cooper pairs are charged ($2e$), they have net zero spin and they obey Bose-Einstein statistics. Their associated energy scale is the gap Δ and gives the energy needed to break one pair. In addition, the characteristic length related to Cooper pairs is the coherence length ξ. Surprisingly, the condensed (phase coherent) ensemble of Cooper pairs in a superconductor is described by a simple wavefunction

$$\Psi = \sqrt{n}\exp(i\phi). \tag{1.1}$$

Here n is the superfluid density (also called density of superconducting carriers) and ϕ the phase of the condensate. It is worth mentioning that $|\Psi|^2$ serves as the order parameter associated with the second order phase transition from the normal to the superconducting state according to the Ginzburg-Landau (GL) phenomenology [4].

Conventional s-wave superconductors described by BCS theory have an isotropic gap structure in momentum space and their Cooper pair size is of the order of 100

[1]The boiling point of helium is 4.1 K

[2]The importance of the discovery is reflected in Richard Feynman's famous article *There's Plenty of Room at the Bottom*. He writes: *"I imagine experimental physicists must often look with envy at men like Kamerlingh Onnes, who discovered a field like low temperature, which seems to be bottomless and in which one can go down and down."*

nm. Consequently, the BCS superfluid density exhibits a *homogeneous* character. *However there also exist superconductors that are radically different*.

Electron pairs in unconventional superconductors

In 1986 a new era for superconductivity began; high-T_C superconductivity was discovered in a cuprate layered material by Bednorz and Muller [6]. Cuprates together with iron-based, heavy fermion and organic superconductors form the family of *unconventional superconductors* that has been the focus of extensive research for the past 35 year in condensed matter physics.

Unconventional superconductors are radically different compared to conventional ones, often involving rich physics. Perhaps the most fundamental difference is the fact that they are made of strongly correlated electrons. These correlations result into additional diverse electronic phases of matter (i.e. Mott phase, pseudogap phase, strange-metal phase, Fermi liquid phase, paramagnetic phase) altogether forming rich temperature versus doping phase diagrams that are mysterious to scientists [7].

Even though the basic idea of Cooper pair formation is also valid for unconventional superconductors, BCS theory fails, among others, to describe their pairing mechanism. For example, the gap in cuprate superconductors is d-wave symmetric whereas for the iron-based superconductor that we study later in this thesis it has been suggested to be $s_{\pm}$-wave. Even though there has been great effort and progress towards understanding the rich physics of these materials, mysteries like the pairing mechanism or the origin of the pseudogap state are still unresolved.

As one may expect, the properties of the Cooper pairs in unconventional superconductors are in stark contrast to conventional superconductors. In unconventional superconductors chemical disorder is present, the coherence length is shorter (a few nm) and the superfluid density is smaller than in conventional ones. The latter is of great importance as it has been shown [8] that the smaller the superfluid density, the more susceptible the superconductor is against fluctuation effects. If Cooper pairs in unconventional superconductors are prone to fluctuations how is in turn their superfluid density affected? Would it still exhibit a homogeneous behaviour similar to conventional superconductors?

Motivated by these considerations, in this thesis among others, we aim to investigate the behaviour of the superfluid density in the atomic scale in an iron-based superconductor. Our ultimate goal is to directly visualize the superfluid.

In the following we shall briefly discuss the core ideas of the method that we employ for probing the superfluid; the Josephson scanning tunneling microscopy. In depth discussion about the working principle of the method is given in Chapter 2.

1.1.1. Imaging the superfluid using Josephson microscopy

In order to be able to visualize the superfluid in the nano-scale we need to employ a method that allows to probe the its Cooper pairs with very high spatial resolution. Towards this end we use the imaging and spectroscopic capabillities of scanning tunneling microscopy (STM) that have been proven of great value in revealing spa-

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tial inhomogeneities in quantum materials [7, 9, 10]. However, as we will see in more detail in Chapter 2, in conventional STM one uses a sharp metallic probe that only addresses the quasi-particles of the material surface under consideration. This prohibits the investigation of the superfluid, unless a *superconducting tip* is employed.

In the heart of STM with a superconducting tip lies the Josephson effect [11] which dictates that Cooper pairs tunnel in a junction formed by two superconducting electrodes driven by the phase difference $\Delta\phi$ between them. Josephson found that the current in a hybrid superconductor-insulator-superconductor (SIS) junction, the *supercurrent* (Cooper pair tuneling current) obeys the relation

$$I_{\mathrm{S}} = I_{\mathrm{C}} \sin(\Delta\phi), \tag{1.2}$$

where I_{C} is the maximum critical supercurrent. Hence we see that using the Josephson effect one is able to detect Cooper pair tunneling. These ideas triggered theorists and experimentalists to use a superconducting tip in a STM in order to probe the condensate with unprecedented spatial resolution. The pioneering works of several research groups [12–16] indicated the immense potential of using superconducting tips in a STM, establishing the Josephson scanning tunneling microscopy (JSTM) technique.

The choice of the material to visualize its superfluid density is of equal importance as the technique. So far the JSTM technique has been insightful in conventional [17] and unconventional high-T_{C} cuprates [18]. In our case, we choose an unconventional iron-based superconductor, namely iron selenide telluride Fe(Se,Te), whose Cooper pair size and superfluid density is small and low, respectively. This makes Fe(Se,Te) a perfect candidate for investigating possible inhomogeneities in the spatial distribution of its superfluid.

So far we have introduced the motivation of directly imaging the superfluid by probing it using the JSTM technique. We highlighted that the unconventional properties of the Cooper pairs in this iron-based superconductor make it vulnerable to fluctuation effects. However, there can also be other mechanisms perturbing the superfluid in the nanoscale. A particularly interesting one, is the competition between the superfluid and magnetic defects. Let us see what governs this interaction and our incentive to bring a superconducting STM tip on Fe(Se,Te) in an attempt to shed more light on it.

The local effect of impurities on the superfluid

Superconductivity and magnetism are two phenomena that compete with each other. External magnetic fields H cause de-pairing in a superconducting material and ultimately destroy any superconducting correlations at sufficiently high fields. The pioneering work of Abrikosov (Nobel prize 2003) [3] using GL theory taught us that there are type I and type II superconductors.[3] However, this and similar works lack any physical consensus for the interaction in the atomic scale. *How is*

[3] In type I superconductors there is no magnetic field penetration in its interior (Meissner effect) up to the critical field H_{C} where the normal state is retrieved. In contrast, type II superconductors exhibit an intermediate state for $H_{\mathrm{C1}} < H < H_{\mathrm{C2}}$, where magnetic fields penetrate inside them in the form of

the superfluid disturbed in the presence of an atomic-sized impurity on its surface carrying a magnetic moment?

The physics involved in the aforementioned interaction is very rich and of great scientific importance in many aspects. It can illuminate the details associated to electronic correlations and result into novel electronic states [19], in both conventional and unconventional superconductors. For example, concerning the question we posed earlier, depending on the strength of the magnetic moment, the local ground state of the system can be either a Kondo-like (locally the moment is screened by the surrounding Cooper pairs) or superconducting-like (the moment remains unscreened) [20]. In this context, Yu, Shiba and Rusinov (YSR) [21–23] studied possible excitations and they calculated that their energy lies inside the energy gap of the superconductor. The so called YSR bound states have been studied in superconductors with great success [19, 24]. Perhaps the most famous examples being the detection of Majorana fermions: (i) in chains of magnetic adatoms placed on Pb [25] (ii) at magnetic impurities on the surface of Fe(Se,Te) [26].

Triggered by the fascinating physics involved, we bring our superconducting tip on the Fe(Se,Te) surface hosting buried impurities. Here, our aim is to investigate how the YSR bound states are influenced by the presence of the low-density superfluid. The use of the superconducting tip allows us to probe the YSR impurity states with unprecedented energy resolution, overcoming the limitations of thermal broadening. Combined with high spatial resolution imaging we address the extent of the YSR resonances in space and comment on their resemblance with the recently detected Majorana bound states [26, 27].

Up to this point the concepts of visualizing the superfluid in Fe(Se,Te) in the nanoscale have been introduced and focus on JSTM, the tool that we will be using in order to probe Cooper pairs, has been given. We envisioned that the superfluid in Fe(Se,Te) possibly exhibits a spatial distribution that differs from conventional superconductors owing to its low density that makes it prone to fluctuations. Furthermore, the motivation to investigate magnetic impurity effects on the atomic scale in the vicinity of this low carrier density condensate has been presented. Next, we want to focus on the importance of detecting another fundamental physical property of the superfluid constituents; their charge. The STM tool used that serves this purpose will be introduced as well.

1.1.2. Charge detection and noise microscopy

We have seen that superconductors consist of charged Cooper pairs that are condensed in a macroscopic quantum state, the superfluid. The charge of a Cooper pair is twice the electron charge $2e$ as a direct consequence of the many-body electronic correlations present in a superconductor. Besides that, Cooper-pairs tunnel across a junction between two superconductors as predicted by Josephson, effectively transferring charge of $2e$ through the junction. Interestingly at higher energies in such junctions, even higher multiples of the electron charge can be transported via multiple Andreev reflections [28]. It thus becomes apparent that detecting the

vortices which we call Abrikosov vortices. For $H < H_{C1}$ they develop the Meissner effect similar to type I superconductors and for $H > H_{C2}$ they become normal.

charge in a superconductor can not only reveal its electronic correlations but also contribute to the understanding of transport mechanisms involving Cooper pairs. Hence the question arises. *How can one measure the charge in a superconductor?* The answer we propose in this thesis lies in a quantity that is usually disregarded in relevant experiments. The *shot noise.*

Electronic shot noise deals with dynamic fluctuations originating from the discrete nature of the electric charge [29]. In contrast to time-averaged current or conductance measurements, shot noise can be employed to obtain information such as the charge and statistics of the quasiparticles involved in transport processes. The way to identify the charge of the transport carriers or possible correlated transport involved is by examining the so called Fano factor $\mathcal{F}$. It is defined as

$$\mathcal{F} = \frac{S}{S_{\mathrm{P}}}, \tag{1.3}$$

where S is the shot noise and $S_{\mathrm{P}} = 2e|I|$ is the shot noise when current, I, of uncorrelated electrons with charge e, flows. When $\mathcal{F} = 1$ the flow consists of independent carriers and the shot noise is equal to the Poisson value S_{P}. On the other hand, there can be systems where the charge of the carriers differs from $1e$ or the carrier flow is correlated. In such cases, $\mathcal{F} \neq 1$, while the time-averaged current or conductance will not be altered.

Shot noise measurements have been proven to be of great value in many instances. Detection of fractional charge in quantum point contacts hosting fractional Hall edge states [30], shot noise doubling in hybrid superconducting junctions due to Andreev processes [31] and enhancement of shot noise in quantum dots [32] are only a handful of examples.

Triggered by the insight that one obtains via shot noise, our goal is to use an STM to measure shot noise as a tool to detect the charge and examine electron correlations in topical superconductors. Towards this end we use noise scanning tunneling microscopy (NSTM) that brings shot noise measurements in the atomic scale. This recently developed microscope [33] (whose working principle and challenges will be analysed in Chapter 2) has already shown great potential in understanding charge trapping in a cuprate superconductor [34], paving the way for future challenging experiments.

As a first step we will be measuring the charge transferred by Cooper pairs through a STM junction formed by conventional superconductors. In such superconducting junctions quasiparticles are retro-reflected as holes effectively transferring Cooper pairs of charge $2e$ across the junction, which we aim to detect. These Andreev reflection processes occur only at specific energy ranges thus a change of the $\mathcal{F}$-factor as a function of energy is anticipated. Last but not least, by mapping the shot noise in the atomic scale around nanocavities we will look how pairing is influenced by chemical disorder.

Our second NSTM endeavour concerns the investigation of the charge of uncondensed carriers in superconductors. In this quest we examine the charge state of a superconductor above T_{C}. We focus on TiN that belongs to a class of superconductors that has not been discussed so far.

Electron pairs in disordered superconductors

Among the different means that destroy superconductivity[4], disorder is perhaps the one that has attracted the most attention over the years. Starting from the early days of BCS theory of superconductivity there has been keen interest on examining what is the effect of disorder on the condensate. Astonishingly, Anderson [35] verified some early experiments by showing theoretically that up to a certain degree of disorder the properties of conventional superconductors remain unaffected.

However it soon became clear that the so called *disordered superconductors* lose their superconducting properties at a critical disorder level often resulting into an insulator or a metal. Consequently, a vast amount of scientific research has focused on the different pathways [36] that destroy the order parameter $|\Psi|$ in low-dimensional disordered superconductors[5]. The early understanding of the mechanism that suppresses superconductivity till breakdown, boils down to the phase and amplitude scenarios, where phase fluctuations or Coulomb repulsion drive the destruction, respectively. Nevertheless, experiments have indicated that the aforementioned division is not always applicable. In particular, localization effects and spatial inhomogeneities of the superconducting properties induced by disorder have pointed towards a picture that contains both scenarios.

In the case of disorder-induced inhomogeneities, low temperature STM has been a key probe towards imaging those. Relevant experiments on disordered superconductors such as TiN [37] and NbN [38] show that close to the critical disorder granular superconductivity emergrges; there exist regions exhibiting superconducting correlations embedded in a matrix where the order parameter $|\Psi|$ is zero.

Notably, STM on strongly disordered superconductors has also suggested the existence of a state that shares a lot of similarities with cuprate superconductors; the *pseudogap state*. In such a state, preformed Cooper pairs without phase coherence exist above T_C and condense below the transition point. This local Cooper pair formation can be attributed to spatial fluctuations of the attractive interaction and leads to a gapped spectrum above T_C as measured on InO [39], TiN [40] and NbN [41].

The idea of phase-incoherent Cooper pairs has motivated us to further investigate disordered superconductors above T_C by means of NSTM. In an attempt to corroborate the possibility of pairing without long-range coherence we directly measure the charge of the carriers above T_C on TiN using shot noise measurements. By employing NSTM we overcome the experimental challenge of discerning between paired and single electrons.

1.2. Thesis outline

The rest of this thesis is organized as follows:

- Chapter 2 is dedicated to the theory aspects of the two major STM techniques that were used in the research work of this thesis. We will analyze in more

[4] Apart from the obvious ones such as temperature and magnetic field, here we refer to intrinsic material properties such as electron density, dimensionality etc.

[5] In lower dimensions, fluctuations are crucial, leading to suppression of the superfluid density

detail the principles of Josephson scanning tunneling microscopy (JSTM) and noise scanning tunneling microscopy (NSTM).

- Chapter 3 summarizes our findings when employing the JSTM technique on the unconventional iron-based superconductor Fe(Te,Se). It is found that the superfluid density in the nanoscale exhibits spatial inhomogeneities associated with a length scale that is comparable to the coherence length.
- Chapter 4 is about the discovery of Yu-Shiba-Rusinov impurity states in Fe(Te,Se) that disperse spatially. This surprising observation is explained by the electric field of the tip that tunes the chemical potential on the impurity resulting into gating. To further corroborate this newly introduced mechanism we performed simulations using the single impurity Anderson model.
- In Chapter 5 shot noise measurements are performed on the conventional superconductor Pb using a Pb STM tip. In this study we combined the aforementioned techniques of Chapter 2 to observe noise doubling as a function of bias voltage due to Andreev reflection processes. Last but not least, in this experiment we map the noise in the nanoscale including sub-surface Ar nanocavities.
- Chapter 6 involves noise spectroscopy on the disordered superconductor TiN, using a metallic Pt/Ir tip. In addition to voltage dependent shot noise curves, we also measure noise at different temperatures (higher than the T_C of TiN). Importantly it is found that noise doubling in TiN due to Andreev reflections persists at temperatures larger than T_C. To conclude, we discuss possible implications of our observations on the current understanding for disordered superconductors.
- Chapter 7 is the last one of this thesis and gives some concluding thoughts together with an outlook for future experiments.

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