Doctoral Thesis

High- and low-temperature superconductors
\(\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8\delta\) and \(2\text{H-NbSe}_2\) as model materials

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High- and Low-Temperature Superconductors: Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ and 2H-NbSe$_2$ as Model Materials

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3.2.1 Isothermal Measurements ................................................. 45
3.2.2 Non-Isothermal Measurements ........................................ 47
3.3 The Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ Compound .............................. 48
3.4 The 2$H$-NbSe$_2$ Compound .............................................. 51

4 Low Magnetic Field Diagram of Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ 55
  4.1 Introduction ............................................................. 55
  4.2 AC-Susceptibility Measurements ....................................... 56
    4.2.1 $\chi_{ac}(T)$-Temperature Sweeps .................................. 57
    4.2.2 $\chi_{ac}(H)$-Field Sweeps ....................................... 61
  4.3 DC-Magnetization Measurements ........................................ 70
    4.3.1 $M_{dc}(T)$-Temperature sweeps ................................... 70
    4.3.2 $M_{dc}(H)$-Field sweeps ......................................... 74
  4.4 Low Field $H$-$T$ Diagram of Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ ........... 78

5 Low Magnetic Field Diagram of 2$H$-NbSe$_2$ 85
  5.1 Introduction ............................................................. 85
  5.2 AC-Susceptibility Measurements ....................................... 86
    5.2.1 $\chi_{ac}(T)$-Temperature Sweeps .................................. 87
    5.2.2 $\chi_{ac}(H)$-Field Sweeps ....................................... 91
  5.3 DC-Magnetization Measurements ........................................ 95
    5.3.1 $M_{dc}(T)$-Temperature Sweeps ................................... 95
    5.3.2 $M_{dc}(H)$-Field Sweeps ......................................... 102
  5.4 Time Relaxation of the non-Equilibrium Vortex State ............... 104
    5.4.1 Intermediate Temperature Regime ($0.52 \leq T/T_c \leq 0.83$) .... 104
    5.4.2 High Temperature Regime ($T/T_c \geq 0.83$) ..................... 108
  5.5 Low Field $H$-$T$ Diagram of 2$H$-NbSe$_2$ .................................. 111

6 Overview and Future Prospects 115

Bibliography 125

Acknowledgments 133

Curriculum vitae 135

List of Publications 137
Abstract

In this thesis, the low field vortex matter phase diagram of the high temperature superconductor Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ has been investigated. For this purpose, susceptibility and magnetization measurements have been performed using a custom made rf superconducting quantum interference device (rf-SQUID) magnetometer. Field sweep as well as temperature sweep measurements have been carried out in a wide range of temperatures 10 K $\leq T \leq$ 100 K and magnetic fields 0 $\leq H \leq$ 800 Oe, with the ac- and the dc-field applied perpendicular to the superconducting CuO$_2$ layers.

The sensitivity and versatility of our experimental arrangement and the high quality of the Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ single crystal ($T_c \approx$ 93 K) allowed us to detect on the same sample all the phase transitions and crossovers which have been observed until now on this high temperature superconductor: the upper critical field [1], the field of first flux penetration [2], the irreversibility transition [3, 4], the melting transition [5, 6], the second peak transition [6, 7], the depinning transition [8, 9] and the zero dimensional crossover [10, 11].

In addition to these transitions, we found a first experimental indication of a crossover related to a change in behaviour of the current density in the low field regime between the first order melting transition and the field of first flux penetration. This crossover field is almost temperature independent in the regime 20 K $\leq T < 37$ K while for $T \geq 37$ K it decreases upon increasing temperature. This observation is in very good agreement with the dimensional transition from a single vortex pinning to a vortex bundle pinning regime recently predicted theoretically by Blatter and coworkers [12–14].
Further, the low field properties \((H < 900 \text{ Oe})\) of the anisotropic low temperature superconductor \(2H-NbSe_2\) \((T_c \approx 7.3 \text{ K})\) has been investigated for reduced temperatures \(T/T_c \gtrsim 0.5\). Susceptibility and magnetization measurements have been performed on high quality \(2H-NbSe_2\) samples as a function of temperature and magnetic field with an \(rf\)-SQUID magnetometer. The \(ac\)- and the \(dc\)-field have been applied perpendicular to the \(ab\)-planes of the single crystals. Further, in order to gain a better understanding of the low field phase diagram deduced from these measurements, the time relaxation of the remanent magnetization \(M_{rem}\) has been investigated with a second custom made magnetometer using a \(dc\)-SQUID. These relaxation measurements cover a time window of five decades \(10^{-1} \text{ s} < t < 10^4 \text{ s}\), so that the current density \(j\) could be studied from values very close to \(j_c\) down to values considerably smaller than \(j_c\).

For fields \(250 \text{ Oe} \lesssim H \lesssim 880 \text{ Oe}\) the temperature sweep susceptibility \(\chi(T)\) and magnetization \(M(T)\) measurements show a feature slightly below the upper critical field \(H_{c2}(T)\). This feature is related to an abrupt increase of the critical current density \(j_c\) known in the literature as peak effect \([15-18]\). For \(H < 250 \text{ Oe}\), this peak effect is no more detectable with our low amplitude susceptibility data, while it is still clearly observed in the magnetization measurements. In the field regime \(85 \text{ Oe} \lesssim H \lesssim 250 \text{ Oe}\) the \(M(T)\) data confirm the low field reentrant behaviour of the peak effect proposed recently \([16-18]\). However, according to our results for \(H \lesssim 85 \text{ Oe}\), the peak effect temperature changes again its behaviour and increases with decreasing field. Moreover, our measurements show that the reentrance of the peak effect is not associated with the reentrant vortex liquid phase as suggested by different authors \([16,17]\). A correct interpretation relates the peak effect phenomenon to an order-disorder transition, as proposed very recently by Paltiel et al. \([18]\).

The time relaxation measurements of the remanent magnetization \(M_{rem}\) in the peak effect temperature regime agrees well with the low field vortex matter phase diagram deduced from our magnetization measurements \(M(T)\) in the neighbourhood of \(T_c\). In the temperature regime \(0.52 \lesssim T/T_c \lesssim 0.82\), where the vortex behaviour is no more affected by the peak effect phenomenon, the flux creep activation barrier \(U\) as a function of \(j\) was found to follow a power law behaviour. Moreover, for the critical current density we obtained \(j_c(T=0) \approx 9.10^3 \text{ A/cm}^2\) in good agreement with results of other groups \([15,19,20]\).
Kurzfassung

In dieser Dissertation wurde das H-T-Phasen-Diagramm des Hochtemperatur-Supraleiters Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ bei schwachen Magnetfeldern untersucht. Zu diesem Zweck wurden Suszeptibilitäts- und Magnetisierungs-Messungen mit einem in unserer Gruppe konstruierten 'rf Superconducting Quantum Interference Device' (rf-SQUID) Magnetometer durchgeführt. Messungen in Abhängigkeit des magnetischen Feldes wie auch der Temperatur wurden in einem breiten Temperatur-Bereich 10 K \( \lesssim T \lesssim 100 \) K und Magnetfeld-Bereich 0 \( \leq H \leq 800 \) Oe durchgeführt, wobei das ac- und das dc-Feld senkrecht zu den supraleitenden CuO$_2$-Ebenen angelegt wurden.

Die Empfindlichkeit und Vielseitigkeit unseres Messsystems wie auch die hohe Qualität des Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ Einkristalles (\( T_c = 93 \) K) ermöglichte uns an der gleichen Probe alle Übergänge, welche bis jetzt in diesem stark geschichteten Hochtemperatur-Supraleiter beobachtet wurden, nachzuweisen: das obere kritische Feld [1], das Feld des ersten Flusseindringens [2], den Irreversibilitäts-Übergang [3,4], den Schmelz-Übergang [5,6], den zweiten Peak-Übergang [6,7], den Entkoppelungs-Übergang [8,9] und den null-dimensionalen Übergang [10,11].

Zusätzlich zu diesen Übergängen fanden wir einen ersten experimentellen Hinweis auf einen Übergang, welcher mit einer Änderung des Verhaltens der Stromdichte zwischen dem Feld des ersten Ordnungs-Schmelz-Überganges und dem Feld des ersten Flusseindringens zusammenhängt. Das Feld dieses Überganges ist nahezu temperaturunabhängig im Bereich 20 K \( \lesssim T \lesssim 37 \) K während es für \( T \gtrsim 37 \) K mit zunehmender Temperatur abnimmt. Diese Beobachtung ist in sehr guter Übereinstimmung mit einem dimensionalren Übergang von einem Einzel-Wirbellinie- zu
Kurzfassung

Ferner wurden die schwachen Feld-Eigenschaften (H < 900 Oe) im Temperatur-Bereich T/Tc > 0.5 des anisotropen Tieftemperatur-Supraleiters 2H-NbSe2 (Tc ≈ 7.3 K) untersucht. Suszeptibilitäts- und Magnetisierungs-Messungen wurden an 2H-NbSe2-Proben in Abhängigkeit des magnetischen Feldes und der Temperatur durchgeführt. Das ac- und dc-Feld wurde senkrecht zu den ab-Ebenen der Einkristalle angelegt. Um ein besseres Verständnis des von diesen Messungen hergeleiteten Phasen-Diagramms bei schwachen Feldern zu erhalten, wurde zusätzlich die zeitliche Relaxation der remanenten Magnetisierung Mrem mit einem zweiten von unserer Gruppe konstruierten dc-SQUID-Magnetometer untersucht. Diese Relaxationsmessungen decken ein Zeitfenster von fünf Dekaden 10^-1 s < t < 10^4 s ab, so dass die Stromdichte j für Werte sehr nahe bei der kritischen Stromdichte j_c bis hinunter zu Werten erheblich kleiner als j_c untersucht werden konnten.

Die zeitlichen Relaxations-Messungen der remanenten Magnetisierung $M_{rem}$ im Peak-Effekt-Temperatur-Bereich sind in guter Übereinstimmung mit dem $H-T$-Phasen-Diagramm für schwache Felder, welches in der Nähe von $T_c$ aus unseren Magnetisierungs-Messungen $M(T)$ hergeleitet wurde. In dem Temperatur-Regime $0.52 \lesssim T/T_c \lesssim 0.82$, wo das Verhalten der Wirbellinien nicht mehr vom Peak-Effekt-Phänomen beeinflusst wird, weist die Aktivierungsbarriere $U$ bezüglich $j$ eine algebraische Abhängigkeit auf. Zudem erhielten wir für die kritische Stromdichte $j_c(T = 0) \simeq 9.10^3$ A/cm² in guter Übereinstimmung mit Werten anderer Gruppen [15, 19, 20].
1 Introduction

Vortices in superconductors are amazing objects. The prediction of quantized flux lines (vortices) in type II superconductors and of their arrangement in a regular lattice was made by Abrikosov [21] in 1957. Since that time many research efforts have been directed to the study of the properties of vortices and vortex lattices. Nevertheless, many questions concerning vortex behaviour in real materials still remain to be answered.

The most simple model of a vortex line in a superconductor is a field “tube” carrying one quantum $\Phi_0 = \frac{hc}{2e}$ of magnetic flux and set up by the shielding currents. When many vortices are present in the sample, their mutual interaction lead to the establishment of an energetically favorable arrangement. Calculations showed that such an arrangement should be a triangular lattice [22]. The first experimental confirmation of this prediction was made in 1964 by neutron diffraction on superconducting Nb [23]. However, it soon became clear that the vortex arrangement in real samples is affected by the internal material structure and inhomogeneities, sample geometry, temperature, field strength, etc. and therefore can be very different from that predicted by Abrikosov’s solution. Most peculiar vortex lattices have been predicted and observed after the discovery of the high temperature superconductors (HTSC) [24]. Experimental and theoretical investigations of the vortex properties in the new materials during the last decade revealed a variety of structural and thermodynamic phase transitions that the vortex lattice can undergo. A completely new concept of “vortex matter”, which is in many ways analogous to “ordinary matter”, arose from these studies. Vortex matter is characterized by a finite elasticity and deforms in response to intrinsic (pinning) or external (current)
forces. When elastic deformation overcomes a certain limit, it deforms plastically via formation of topological defects in the vortex lattice. If temperature increases vortices undergo thermal displacements just like atoms in a solid, and above certain temperatures, the lattice melts into a "liquid" of vortex lines which are almost free to move. Moreover, in layered materials with highly anisotropic superconducting properties vortex lines passing through the layers can decouple and evaporate into a "gas" of vortex pancakes.

In spite of the intense experimental and theoretical work of the last years, the complicated vortex matter phase diagram of high temperature superconductors is far from being completely elucidated. The exact nature of the vortex structure in the several different vortex phases is actually not fully known and there is no general consensus about some type of transitions between the different vortex regions. The main focus of our work is to investigate the low field $H$-$T$ phase diagram of a high temperature superconductor using an ultra sensitive non commercial rf-SQUID-magnetometer and a high quality Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ single crystal. Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ is the paramount example of HTSC showing the relevant characteristics associated with the properties of high temperature superconductors: high anisotropy, elevated critical temperature, and weak pinning. For the investigations of the low field vortex behaviour, susceptibility and magnetization measurements have been performed with the ac- and the dc-field applied perpendicular to the $ab$-planes of the single crystal. The sensitivity of our experimental arrangement allows us to reproduce all known phase transitions and crossovers on a single sample and compare them with the respective theoretical models. Moreover, we have found some indication for a new transition in the field regime between the melting transition and the field of first flux penetration. This transition is in good agreement with the crossover from a single vortex pinning regime to a vortex bundle pinning regime recently predicted by Blatter and coworkers [12–14].

In high temperature superconductors the combination of high operating temperatures, short superconducting coherence length and high anisotropy enormously enhances the role of thermal fluctuations of the flux lines. The most important effect of these thermal fluctuations is the melting of the vortex lattice observed at temperatures well below the upper critical field $H_{c2}(T)$. The resulting vortex liquid phase has been found to occupy a significant upper part of the $H$-$T$ phase diagram.
1. Introduction

A particularly interesting theoretical prediction in anisotropic and layered superconductors is also the existence of a narrow vortex liquid phase just above the lower critical field $H_{c1}(T)$, first proposed by Nelson [25] and further elaborated in numerous other theoretical studies [26, 27]. However, high temperature superconductors are not ideal candidates for the detection of this low field melting line, since the width of the narrow vortex liquid phase in these materials is predicted to be a few Oersted [26]. Better candidates are the weak pinning, short penetration length systems, such as the anisotropic low temperature superconductor $2H$-NbSe$_2$, where the width of the low field vortex liquid phase is estimated to be of about twelve Oersted.

The anisotropic superconductor niobium diselenide $2H$-NbSe$_2$ has attracted a great deal of attention largely because many of its properties lie in between those of conventional superconductors and the cuprates. In the last few years several results at low magnetic fields on $2H$-NbSe$_2$ single crystals have been presented [16, 28] showing an anomalous peak in the critical current density $j_c$ known as ‘peak effect’ (PE) slightly below the upper critical field $H_{c2}(T)$. Moreover, for magnetic fields $50 \text{ Oe} \lesssim H \lesssim 150 \text{ Oe}$ a weak reentrant behaviour of the PE phenomenon has been observed recently [16, 17]. Relying on the assumption that the peak effect can be associated with the melting of the vortex lattice [29, 30], this reentrant behaviour has been interpreted as a first experimental indication of the predicted vortex liquid phase at low magnetic fields [16, 17]. However, for magnetic fields $H \lesssim 50 \text{ Oe}$ the PE was no more detectable [16, 17]. The goal of our work has been to investigate the peak effect with the emphasis on fields $H \lesssim 50 \text{ Oe}$ in order to determine the reentrant behaviour and the relation between the PE phenomenon and melting of the flux line lattice in the field regime very close to $H_{c1}(T)$. For this purpose, susceptibility and magnetization measurements with our custom made $rf$-SQUID-magnetometer have been performed on high quality $2H$-NbSe$_2$ single crystals. In order to gain a better understanding of the low field phase diagram deduced from these measurements, the time relaxation of the remanent magnetization $M_{rem}$ in the reduced temperature regime $0.5 \lesssim T/T_c < 1$ has been further investigated with a non commercial $dc$-SQUID-magnetometer. Our investigations confirm the low field reentrant behaviour of the peak effect temperature [16, 17] in the field regime $85 \text{ Oe} \lesssim H \lesssim 250 \text{ Oe}$. However, according to our results, for fields $H \lesssim 85 \text{ Oe}$ the behaviour of the peak effect temperature changes, increasing upon decreasing field.
Moreover, our measurements show that the reentrance of the peak effect is not related to the reentrant vortex liquid phase. A better explanation is given associating the peak effect phenomenon to an order-disorder transition as proposed recently [18].

The thesis is organized as follows:

- The aim of Chapter 2 is to introduce the general theoretical concepts and tools used to describe vortices subject to thermal fluctuations and to quenched disorder in layered type II superconductors. The main transitions in the phase diagram and in the pinning diagram are theoretically discussed.

- Chapter 3 describes the experimental arrangement and the measuring procedure used for the investigations. Furthermore, it gives a brief description of the Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ compound, the 2H-NbSe$_2$ compound and the measured specimens.

- Chapter 4 presents susceptibility and magnetization measurements performed on a Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ single crystal for magnetic fields applied perpendicular to the superconducting CuO$_2$ layers. The low-field magnetic diagram obtained from the measurements is compared with reports from literature and discussed within theoretical models described in Chapter 2. A new crossover between the first order melting transition field and the field of first flux penetration is proposed.

- Chapter 5 reports about susceptibility, magnetization and time relaxation measurements on a 2H-NbSe$_2$ single crystal for magnetic fields applied parallel to the crystal c-axis. The measurements are compared with recently published results and a qualitative phase diagram of the low-field high-temperature vortex state in 2H-NbSe$_2$ is proposed.
2 Vortex Properties in Layered Type II Superconductors

2.1 Introduction

Superconductivity is phenomenologically well described by the Ginzburg-Landau (GL) theory which is based on a free-energy functional describing superconductivity in terms of a complex order parameter $\Psi(r)$ coupled to the electromagnetic vector potential $A$. The order parameter can be interpreted as the macroscopic wave function of the condensate of Cooper pairs and takes the simple form $\Psi = |\Psi| \exp(i\phi)$ for s-wave superconductors which we will assume in this chapter. The London theory can be seen as a special case where $\Psi(r) = \text{const.}$ all over the sample. The GL functional contains two relevant length scales: The magnetic penetration length $\lambda$ which is the typical scale over which the vector potential $A$ varies, and the coherence length $\xi$ which represents the correlation length of the Cooper pairs and gives the scale for variations of the order parameter $\Psi$. The mean-field treatment of the GL free energy leads to the common classification in two types of superconductors, depending on the ratio $\kappa = \lambda/\xi$ (Ginzburg-Landau parameter).

If $\kappa < 1/\sqrt{2}$, the superconductor is called type I and the phase diagram contains only two phases: At magnetic fields $H < H_c(T)$ and temperatures $T < T_c$ the sample is in the superconducting Meissner-Ochsenfeld state where the magnetic field is completely expelled, i.e., the magnetic induction $B = \nabla \wedge A = 0$ inside the probe (perfect diamagnetism). In this superconducting phase all the electrons are paired into Cooper pairs, resulting in $|\Psi(r)| = \text{const.}$ and zero electric resistivity. In the normal phase $H > H_c(T)$ the diamagnetic property is lost; the order parameter
2. Vortex Properties in Layered Type II Superconductors

\( \Psi(r) = 0 \) everywhere and the resistivity is finite.

If on the other hand \( \kappa > 1/\sqrt{2} \) the superconductor is called type II. In addition to the Meissner-Ochsenfeld phase \( (B = 0) \) below the so called lower critical field \( H_{c1}(T) \), and the normal phase \( (B = H) \) above the upper critical field \( H_{c2}(T) \), one has the vortex phase \( (0 < B < H) \) at intermediate fields \( H_{c1}(T) < H < H_{c2}(T) \).

This mixed phase, also called Shubnikov phase, is characterized by the fact that the magnetic field penetrates the sample in the form of flux tubes called vortices, each carrying a flux quantum \( \Phi_0 = \frac{hc}{2e} \approx 2 \cdot 10^{-7} \text{ Gcm}^2 \). These vortices consist of a normal-conducting core region of extent \( \xi \), encircled by superconducting currents. Vortices repeal each other due to the electromagnetic interaction, which have a range \( \lambda \) and arrange themselves into a triangular lattice named after Abrikosov who predicted this phenomenon in 1957.

Due to the large extension of the mixed phase in the high \( T_c \) superconductors \( (H_{c1} \sim 10^{-2} \text{T}, \ H_{c2} \sim 10^2 \text{T}) \) and the fact, that vortices can be compared to interacting elastic strings, the behaviour of vortices in the mixed phase has been the subject of extended experimental and theoretical research in the last decade. Further, investigations of the vortex state are important for technological applications since an applied electrical current causes the vortices to move and this in turn leads to dissipation, i.e., a nonzero resistivity. To be of technological use, the motion of vortices must be prevented. This is practically achieved by material defects, which "pin" the flux lines and give rise to a critical current density "\( j_c \)" below which transport currents flow practically without dissipation. Nevertheless the resistivity is not completely vanishing, due to the fact that the vortices can move out of pinning centers due to thermal activation. However it turns out that the barriers \( U_c \) for this creep motion are large for \( j < j_c \), reducing the resistivity exponentially. Understanding the pinning problem is thus of great importance with regard to practical applications.

Although the phenomenology of the vortex system has been extensively investigated, at this time there is still no general consensus about the structure of the mixed phase in the \( B-T \) diagram. This \( B-T \) diagram is usually summarized in two different types: The first one is the phase diagram which shows the various thermodynamic phases of vortex matter at thermal equilibrium. The second describes the pinning properties of the disordered vortex lattice and is accordingly called pinning diagram. The
various regimes in this diagram specify the nature of collectively pinned domains in the vortex matter, which are relevant in the determination of the critical current density $j_c$ and the activation barrier $U_c$ for vortex creep.

The aim of this chapter is to introduce some general theoretical concepts and tools used to describe vortices subject to thermal fluctuations and to quenched disorder as material defects in layered type II superconductors. In section 2.2 we briefly present the phenomenological description of continuous anisotropic (e.g. $2H$-NbSe$_2$) and discrete layered (e.g. $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$) type II superconductors. Section 2.3 deals with the effects of thermal fluctuations on the vortex lattice and describes the main transitions in the phase diagram. Section 2.4 treats the effects of quenched disorder on the vortex lattice and a low field pinning diagram of layered superconductors proposed recently by Wagner, Blatter et al. [12–14] is discussed.

2.2 Phenomenological Description of Layered Superconductors

In the case of anisotropic layered materials, it appears that the Ginzburg-Landau theory must be adapted to include the layered structure explicitly. For not too large anisotropy, a description in terms of a continuous anisotropic Ginzburg-Landau or London theory is applicable. On the other hand, for very large anisotropy the discreteness of the structure becomes relevant, and a description in terms of a set of weakly coupled superconducting layers is more appropriate. Such a description is given by the discrete Lawrence-Doniach model [31], which provides the basis for the discussion of the physics of layered superconductors.

The criterion usually adopted to go from a continuous anisotropic to a discrete layered description is the smallness of the coherence length $\xi_c$ along the $c$-axis with respect to the layer separation $d$ as expressed by the dimensionless ratio $\tau_{\text{cr}} = 2\xi_c^2/d^2$ [26]. The ratio $\tau_{\text{cr}}$ characterizes the crossover from 3D anisotropic to quasi-2D layered behaviour: For a large coherence length $\xi_c$, i.e. $\tau_{\text{cr}} \gg 1$ the continuous anisotropic description is always appropriate. On the other hand, for small $\tau_{\text{cr}} \ll 1$ a crossover will take place at a temperature $T_{\text{cr}} = (1 - \tau_{\text{cr}})T_c$ where
the system behaves in a quasi 2D manner at low temperatures, and exhibits 3D anisotropic behaviour above \( T_c \). In the case of the low temperature superconductor \( 2H-NbSe_2 \) \( (\tau_c \sim 10) \) and the high temperature superconducting \( Y-Ba-Cu-O \) compound \( (\tau_c \sim 5) \) a continuous anisotropic description is applicable, whereas the more strongly layered superconductor \( Bi_2Sr_2CaCu_2O_{8+\delta} \) belongs to the class of materials with \( \tau_c \sim 10^{-3} \ll 1 \) and hence the Lawrence-Doniach model applies.

It should be pointed out that the continuous anisotropic Ginzburg-Landau or London-based analysis often provides a rather good description of the physics of layered materials, for example, for the discussion of the elastic properties of the vortex lattice. Other properties, e.g., the thermodynamic properties of the superfluid or of the vortex lattice, resemble more closely the properties of a 2D superconducting film than those of a 3D bulk material, and consequently are better described by the Lawrence-Doniach model. The question of the continuous anisotropic description versus the discrete layered description therefore not only has to be decided by the size of the coherence length \( \xi_c \) with respect to the layer spacing \( d \), but depends also strongly on the specific physical question at hand [26].

In the following, the anisotropic Ginzburg-Landau theory and the discrete layered Lawrence-Doniach model introduced above are shortly described. We restrict ourself to the special case where the applied magnetic field \( H \) is parallel to the symmetry axis \( (c\text{-axis}) \).

### 2.2.1 Anisotropic Ginzburg-Landau Theory

The basic frame for the phenomenological description of superconductivity is given by the Ginzburg-Landau (GL) free-energy functional

\[
\mathcal{F} = \int d^3r \left( \alpha |\Psi|^2 + \frac{\beta}{2} |\Psi|^4 + \sum_{i=1}^3 \frac{1}{2m_i} \left( \frac{\hbar}{i} \frac{d}{dx_i} + \frac{2e}{c} A_i \right) \Psi \right)^2 + \frac{B^2}{8\pi} - \frac{HB}{4\pi}, \tag{2.1}
\]

where \( \Psi(r) \) is the order parameter, \( A \) is the vector potential, \( B = \nabla \times A \) is the microscopic magnetic induction, and \( H \) is the applied external field. The GL parameter \( \alpha = -\alpha(0)(1 - T/T_c) \) changes sign at the transition temperature \( T_c \),
whereas $\beta$ is taken to be constant in temperature. The parameters $m_i$ ($i = a, b, c$), denote the effective masses along the main axes of the crystal. When applying this description to layered superconducting materials, the anisotropy of the material has to be taken into account. For the sake of simplicity and because layered materials are within high accuracy uniaxial (axis $\parallel c$) materials, $m_a = m_b = m_{ab}$ are chosen and the mass anisotropy ratio is denoted by

$$\epsilon^2 = \frac{m_{ab}}{m_c} \leq 1.$$  \hfill (2.2)

The mass anisotropy causes the coherence length $\xi$ anisotropy

$$\xi_i^2(T) = \frac{\hbar}{2m_i |\alpha|}.$$  \hfill (2.3)

where the subscript $i$ identifies a particular principal axis. Since $\alpha(T)$ is isotropic and proportional to $(T - T_c)$, $\xi_i$ scales with $1/\sqrt{m_i}$ and diverges as $|T - T_c|^{-1/2}$. Further, from the relation

$$2\sqrt{2} H_c(T) \xi_i(T) \lambda_i(T) = \Phi_0,$$  \hfill (2.4)

it can be observed that the anisotropy of the penetration depth $\lambda_i$ will be inverse to that of $\xi_i$ since the thermodynamical critical field $H_c$ is isotropic. In applying this rule it has to be remembered that $\lambda_i$ describes the screening by supercurrents flowing along the $i$-th axis, not the screening of a magnetic field along the $i$-axis.

The anisotropy of the GL parameters involves a modification of the structure of a single vortex. If a magnetic field is applied along the $a$-axis of an isotropic superconductor, this vortex would have circular symmetry. In an anisotropic superconductor, the core radius along the $ab$-plane direction will be $\xi_{ab}$, whereas the core radius in the $c$ direction will be $\xi_c \ll \xi_{ab}$. On the other hand, the flux penetration radius will be $\lambda_c$ along the $ab$ plane direction, whereas it will be the smaller value $\lambda_{ab}$ in the $c$-direction. Thus, both the core and the current streamlines confining the flux are flattened into ellipses with long axes parallel to the planes, with aspect ratio $(m_c/m_{ab})^{1/2}$. 
A further consequence of the anisotropy of the GL parameters is the fact, that the critical fields $H_{c1}(T)$ and $H_{c2}(T)$ in anisotropic superconductors become angular dependent, whereby

$$\epsilon = \left(\frac{m_{ab}}{m_c}\right)^{1/2} = \frac{\lambda_{ab}}{\lambda_c} = \frac{\xi_{ab}}{\xi_c} = \frac{H_{c2||ab}}{H_{c2||c}} = \frac{H_{c1||ab}}{H_{c1||c}}.$$  \hspace{1cm} (2.5)

One common way used to determine the GL anisotropy parameter $\epsilon$ of an anisotropic superconductor is to measure the critical field $H_{c2}$ in the direction perpendicular and parallel to the $ab$-plane.

### 2.2.2 Discrete Layered Lawrence-Doniach Model

A convenient model for the analysis of the consequences of a layered structure in a superconducting material was proposed by Lawrence and Doniach [31]. In their model, layered superconductors are viewed as a stacked array of two-dimensional superconductors, within each of which the GL order parameter $\psi_n(x,y)$ is a 2-D function, coupled together by Josephson tunneling between adjacent layers.

As in the conventional GL theory, the Lawrence-Doniach (LD) model is defined in terms of a free-energy expression for the stack of layers. Omitting the vector potential for simplicity, the free energy can be written as

$$\mathcal{F} = \sum_n \int d^2r \left( \alpha \left| \frac{\partial \psi_n}{\partial x} \right|^2 + \frac{\beta}{2} \left| \psi_n \right|^4 + \frac{\hbar^2}{2m_{ab}} \left( \left| \frac{\partial \psi_n}{\partial x} \right|^2 + \left| \frac{\partial \psi_n}{\partial y} \right|^2 \right) + \frac{\hbar^2}{2m_c d^2} \left| \psi_n - \psi_{n-1} \right|^2 \right).$$ \hspace{1cm} (2.6)

In this expression a possible anisotropy in the $ab$ plane has been ignored. Note that if writing $\psi_n = | \psi_n | e^{i \phi_n}$, and assuming that all $| \psi_n |$ are equal, the last term of (2.6) becomes

$$\left( \frac{\hbar^2}{2m_c d^2} \right) | \psi_n |^2 \left[ 1 - \cos(\phi_n - \phi_{n-1}) \right],$$ \hspace{1cm} (2.7)

which makes clear the equivalence of this term to a Josephson coupling energy between adjacent planes [32].
2.2 Phenomenological Description of Layered Superconductors

The LD model predicts, that with the magnetic field $H$ directed perpendicularly to the layers, the vortex lines divide up into loosely coupled strings of so-called pancake vortices (see figure 2.1) [33]. A pancake vortex is a two-dimensional shaped vortex confined into the layer and surrounded by nearly circular currents. These stacked 2D vortices are coupled together by means of Josephson vortices whose axes thread through the Josephson junctions between the superconducting layers, stretching from the center of each pancake vortex to the center of the adjacent vortices above and below. Thermal agitation can shake the stack, decouple pancake vortices in adjacent layers, and even cause the stack to break up.
2.3 Phase Diagram of Layered Type II Superconductors

The phase diagram in a superconductor describes the various thermodynamic phases of vortex matter at equilibrium. Stimulated by the discovery of the high-$T_c$ materials, a reexamination of this mean-field phase diagram unraveled three main new phenomena.

First, it was experimentally and theoretically observed that due to enhanced thermal fluctuations the vortex lattice can melt into a liquid, just like an ordinary solid. Recently very sensitive calorimetric and magnetic measurements have proved this transition to be of first order [5, 34]. Further, theoretical results show [26], that the position of the melting line in the $B$-$T$ diagram depends strongly on the elastic properties of the vortex lattice and on material parameters, as for example the critical temperature $T_c$. While for low $T_c$ materials the melting line is predicted to lie close to the upper critical field $H_{c2}(T)$, for high temperature superconductors the melting transition lies well below the superconducting to normal state crossover.

Second, pointlike disorder such as pinning-centers, can have an important impact upon the equilibrium field-temperature phase diagram. It is argued that [35-38] in the solid vortex phase, pointlike disorder can produce a weakly disordered glassy state. While, at high temperature and fields the melting of this vortex glassy state into a liquid phase is nearly insensitive to disorder [39], by increasing the field at low temperature disorder is able to make vortex dislocations proliferate, destroying the glassy state [39]. The phase into which the glassy state “melts” at low temperatures is poorly understood and a subject of theoretical investigations [39-41].

Third, the presence of surface [42,43] and geometrical barriers [44] inhibit the vortex entry into the superconductor, while they do not act significantly against their exit. In relatively clean systems these surface effects will be the dominant sources of the experimentally observed irreversibility line (IL) in the $H$-$T$ phase diagram, that separates the reversible magnetization at high temperatures from hysteretic behaviour at lower temperatures.

In this section the influence of thermal fluctuations on the vortex lattice are treated. More precisely, in section 2.3.1, solutions for the melting line derived theoretically for the two regimes $H_{c1} \ll B \ll H_{c2}$ and $H_{c1} \lesssim B$, where the elastic
properties of the vortex lattice and hence its stability with respect to thermal fluctuations are rather different [45-47], are described. In section 2.3.2 the experimentally observed “second magnetization peak” in high-$T_c$ materials [6,7,48,49] is discussed. There is currently no commonly theoretical accepted explanation for this “transition”. However, two different theoretical approaches are treated, which are in good qualitative agreement with experimental results and are based on the disordered induced transition [39-41]. Finally, in section 2.3.3 we discuss the irreversibility line, the crossover-line in the $B$-$T$ diagram above which energy barriers that inhibit the vortex entry into the superconductors disappear.

2.3.1 Melting Line

For the following considerations concerning the theoretical derivation of the melting line for the two regimes $H_{c1} \ll B \ll H_{c2}$ and $B \lesssim H_{c1}$, we will closely follow the approach in [26].

2.3.1.1 $H_{c1} \ll B \ll H_{c2}$ Regime

In this subsection we will concentrate on the intermediate regime characterized by the conditions $H_{c1} \ll B \ll 0.2H_{c2}$. The first inequality guarantees that the mean vortex separation $a_0$ is small compared to the London penetration depth $\lambda$; hence the interaction between the individual vortex lines is large and strongly nonlocal. The second inequality makes sure that the vortex cores do not overlap, and thus the London approximation can be used to determine the energy of the elastically deformed vortex lattice [46].

For high-$\kappa$ materials, the relation $H_{c2} = 2\kappa^2H_{c1}/\ln\kappa$ shows that the regime $H_{c1} \ll B \ll 0.2H_{c2}$ is very large and covers most of the experimentally accessible field range. Unfortunately, even for this well defined situation, no consistent theory of vortex lattice melting is known today. Various approaches have been used to tackle the problem of vortex lattice melting. The two most prominent methods are based on the Lindemann criterion and on Monte Carlo simulations. In the following we will concentrate on the analysis based on the first method.
The semiquantitative approach based on the Lindemann Criterion [50] assumes that a crystalline lattice becomes unstable with respect to thermal fluctuations of its elements as the mean-squared amplitude of fluctuations \( \langle u^2(T_m) \rangle_{th} \) increases beyond a fraction \( c_L \) of the lattice constant \( a_o \),

\[
\langle u^2(T_m) \rangle_{th} \approx c_L^2 a_o^2.
\] (2.8)

With a Lindemann number \( c_L \approx 0.1 - 0.2 \) depending only weakly on the specific material, the criterion (2.8) provides a reasonable estimate of the melting temperature \( T_m \) for a large variety of three-dimensional solid-liquid transitions. For the present discussion of vortex lattice melting, the use of the Lindemann criterion is particularly fruitful, as it allows us to determine the shape of the melting line \( T_m(B) \) over a broad range of magnetic field values by assuming a constant value of \( c_L \) over the entire range \( H_{c1} \ll B \ll 0.2H_{c2} \). As both the type of lattice and the (long-range) nature of the interaction are unchanged throughout this regime, such an assumption seems to be quite reasonable.

In order to make use of the Lindemann criterion (2.8) one has to determine the mean-squared amplitude of fluctuations \( \langle u^2(T_m) \rangle_{th} \) for the vortex lattice. This has been calculated by Houghton et al. [51] and Brandt [52]. For an isotropic material or for the special case with \( H \parallel c \), one has to solve the following implicit equation for the melting line [51]

\[
\frac{\sqrt{b_m(t)}}{1 - b_m(t)} \left( \frac{t}{\sqrt{1 - b_m(t)}} \right) = \frac{2\pi c_L^2}{\sqrt{G_i}},
\] (2.9)

with \( b_m(t) = B_m(T)/H_{c2}(T) \), \( t = T/T_c \) and \( G_i = 10^{-9} \kappa^4 T_c^2/H_{c2}(0) \) the Ginzburg number, which determines the width of the fluctuation region close to the upper critical field \( H_{c2}(T) \). For temperatures close to \( T_c \), the melting line is far below the upper critical field \( H_{c2}(T) \) and the implicit equation (2.9) can be simplified considerably to

\[
B_m(T) = \beta_m \frac{c_L}{G_i} H_{c2}(0) \left( 1 - \frac{T}{T_c} \right)^2,
\] (2.10)
with $\beta_m \approx 5.6$. For discrete layered HTSC, as for example the Bi-Sr-Ca-Cu-O compound, the Ginzburg number is of the order of $G_i \approx 1$, so that the condition $B_m \ll 0.2H_{c2}$ and the result (2.10) is valid over a wide regime in temperature below $T_c$.

For continuous anisotropic HTSC with typical values of $G_i \sim 10^{-2}$, as for example the Y-Ba-Cu-O compound, the result in equation (2.10) is only valid near $T_c$. Away from $T_c$, the melting line moves closer to the $H_{c2}(T)$ line, and the suppression of the order parameter due to overlapping vortex cores becomes relevant. Hence, the full equation (2.9) has to be used for the determination of the melting line. The main correction to the simple power-law result can be obtained by accounting for the suppression of the order parameter via the substitution $\lambda = \lambda' = \lambda/(1 - b(t))^{1/2}$, and the melting line is then given by [26]

$$B_m \approx \begin{cases} \frac{H_{c2}(0)c_4^2\beta_m}{G_i} \left(1 - \frac{T_c}{T}\right)^2 & T \rightarrow T_c \\ \left(1 - \frac{T}{T_c}\right) & (T_c/T - 1) > G_i/\left(\beta_m c_4^4\right) \end{cases}$$

(2.11)

Also the behaviour of the melting line very near the critical temperature $T_c$ cannot be analyzed completely without exploring the properties of the vortex lattice within the fluctuation regime. A discussion for the (scaling) behaviour of the melting line within this regime has been given by Fisher et al. [53]. On approaching the critical temperature $T_c$, the superconductor is expected to enter the regime where the fluctuations in the order parameter $\Psi$ are essentially those of an unchanged superfluid or XY model. Within this regime, the two fundamental length scales $\xi$ and $\tilde{\lambda}$ are expected to scale according to $\xi \sim \xi_0(1-T/T_c)^{-2/3}$ and $\tilde{\lambda} \sim \lambda_0(1-T/T_c)^{-1/3}$ with $\xi/\tilde{\lambda} = (1/2\pi c_4^2(2G_i)^{1/2})\xi/\lambda$. Within the fluctuation dominated regime the temperature behaviour of the melting line is then expected to be $B_m(T) \propto (1-T/T_c)^{4/3}$ [53].

When we combine these different consideration, it does not seem unreasonable that the melting field $B_m(T)$ which should vary as $(1-T/T_c)^{4/3}$ very near $T_c$, then changes over to $(1-T/T_c)^2$ in the mean field region, yet always stays below a curve
Vortex Properties in Layered Type II Superconductors

rising as \((1 - T/T_c)\), will not have a unique power law fit, but will give a reasonable fits to various powers that are intermediate between 1 and 2, depending on the temperature range and the parameters of the material. This is in good agreement with various experimental determinations of the melting line in the high temperature superconductors \(\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}\) and \(\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}\). Whereas Farrell et al. [54] and Beck et al. [55] obtained for \(B_m(T) \propto (1 - T/T_c)^\beta\) an exponent \(\beta \approx 2\), the measurement of Schilling et al. [56], Safar et al. [57] and Zeldov et al. [5] produce power-law exponents in the range 1.35 - 1.55.

2.3.1.2 \(B \lesssim H_{c1}\) Regime

The existence of a vortex liquid phase at very low magnetic inductions \(B \lesssim H_{c1}\) and hence the presence of a melting line just above the onset of the mixed state was first predicted by Nelson in 1988 [25]. The origin of this low field melting transition is found in the weak interaction between the vortex lines at very low magnetic fields. In this section we will give a short theoretical derivation of this low field melting transition \(B_m(T)\). For a more detailed treatment see literature [26,27].

For \(B \lesssim H_{c1}\) the intervortex spacing increases beyond the penetration depth \(\lambda\), and the vortex-vortex interaction decreases exponentially. Due to this dependence of the interaction between the vortex lines in the present low density regime, the applicability of the Lindemann criterion based on a density independent Lindemann number \(c_L\) is much less obvious. However, the approach is still useful for providing an order-of-magnitude estimate for the melting transition.

In order to estimate the mean-squared amplitude of thermal fluctuations \(\langle u^2(T_m)\rangle_{th}\), the appropriate expression for the elastic moduli valid in the low density regime \(B \lesssim H_{c1}\) has to be used. In case of substantial anisotropy the lower branch of the melting line mainly derives from the electromagnetic coupling term in the tilt module and is independent of material anisotropy. The expression of the low field melting line \(B_m(T)\) is then given by [58]:

\[
B_m(T) \approx \frac{\Phi_0}{4\lambda^2} \left[ \ln \left( \frac{4\pi c_L^2}{(3\pi)^{1/4} T} \right) \right]^{-2}
\]

(2.12)
Figure 2.2: Low field melting lines calculated for Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ ($\lambda = 2000$ Å, $\epsilon = 1/100$ and $T_c = 90$ K) and 2H-NbSe$_2$ ($\lambda = 700$ Å, $\epsilon = 1/3$ and $T_c = 7.3$ K) are shown. A temperature dependent penetration depth $\lambda(T) = \lambda_0(1 - (T/T_c)^2)^{1/2}$ and a Lindemann number $c_L = 0.1$ were used in the calculation.

with $\epsilon_o = (\Phi_o/4\pi\lambda)^2$ the basic energy scale of the continuum elastic theory.

Using parameters appropriate for the high temperature superconductors BSCCO and YBCO, the maximum of the low field melting line is found to be of the order of a few Gauss (see figure 2.2). Oxide superconductors with their large values of $\lambda$ are therefore not ideal candidates for the experimental detection of this low vortex liquid phase. Better candidates are the weak pinning, short penetration length system such as for example the anisotropic type II superconductor 2H-NbSe$_2$ [16]. An estimate with equation 2.12 shows that for the parameters of 2H-NbSe$_2$ ($\lambda = 700$ Å, $\epsilon = 1/3$ and $T_c = 7.3$ K) with a temperature dependent penetration depth $\lambda(T) = \lambda_0(1 - (T/T_c)^2)^{1/2}$ and a Lindemann number $c_L = 0.1$, the maximum amplitude of the low field branch of the melting line will be about 12 Oe at about $1/3 \ T_c$ (see figure 2.2).
2.3.2 Second Magnetization Peak Line

An intriguing feature of many high temperature superconductors is the experimental observation of a sudden increase in the critical current density $j_c$ when the field is increased at low temperatures $[6,7,48,49]$. Unlike first order melting, this phenomenon is accompanied by an abrupt but continuous change of the magnetization at a magnetic induction $B_{sp}(H)$, suggesting a second order phase transition or a crossover $[6,59]$. Due to the shape of the magnetization curves when a field is applied in the temperature regime where the increase of $j_c$ is observable, the transition at $B_{sp}(H)$ has been called "second magnetization peak transition". The second magnetization peak line and the melting line in the $B$-$T$ phase diagram are apparently found to form one continuous transition line that changes from first to probably second order at a temperature $T_{cr}$, a so called "multicritical point" $[6,49]$.

There is currently no commonly accepted theoretical explanation for this second magnetization peak line. The associated increase of the magnetization with magnetic field has been attributed to surface barrier effects $[7]$, a crossover from surface barrier to bulk pinning $[43]$, sample inhomogeneities by oxygen deficient regions $[48]$, dynamic effects due to different relaxation rates in single vortex and small-bundle pinning regimes $[60,61]$, the existence of a dimensional crossover field from 3D to a 2D regime $[62]$ or from a 1D to a 3D regime $[10]$, a disorder induced decoupling transition $[6,40]$, and a disorder induced entanglement transition $[39,41]$. As the last two represent the most accepted ideas, we will give a short description of them.

Horovitz and Goldin $[40]$ studied the phase transition of a flux lattice in layered superconductors with magnetic field perpendicular to the layers and in the presence of strong disorder. They find that the Josephson coupling between layers leads to a strongly pinned Josephson Glass phase at low temperatures and fields. In their theory the experimentally observed second magnetization peak line is interpreted as a decoupling transition from this strongly pinned Josephson Glass at low fields to a pinned Glass phase at high fields. This phase transition at $B = B_0$ is determined by the disorder strength and is $T$ independent up to the multicritical point at $T_0 \simeq (\tau a_o^2 \ln(a_o/d))/(8\pi \lambda_{ab}^2)$ where $\tau = \Phi_0^2 d/4\pi^2 \lambda_{ab}^2$, $d$ is the spacing between layers, $\lambda_{ab}$ is the magnetic penetration length parallel to the layers and $a_o = \sqrt{\Phi_0/B_0}$. 


An alternative theoretical approach has been developed by Ertas and Nelson [41]. They applied the Lindemann criterion (equation (2.8)) to a “cage model” [41] and examined the combined effect of point disorder and thermal fluctuations on the vortex lattice. They explain the second magnetization peak as a dislocation induced phase transition from a weakly disordered quasi vortex lattice (Bragg Glass), to a highly disordered entangled glass (entangled vortex Glass). The temperature behaviour of this entanglement transition is predicted as follows [41]:

In the low temperature region \( T < T^* \simeq (\epsilon^2 \epsilon_0 \delta)^{1/3} \), thermal fluctuations are smaller than disorder fluctuations and the entanglement transition field is given by

\[
B_{en}(0) \simeq \frac{\Phi_0}{\xi_{ab}(0)} \left( \frac{\epsilon \epsilon_0 \xi_{ab}(0)}{T^*} \right)^{10/3} c_L^{16/3},
\]

where \( \xi_{ab} \) is the in-plane coherence length, \( \epsilon_0 = (\Phi_0/4\pi \lambda_{ab})^2 \), \( \lambda_{ab} \) the in-plane penetration depth, \( \delta \) the disorder parameter, \( \epsilon \) the anisotropy parameter, and \( c_L \) the Lindemann number.

With increasing temperature, thermal fluctuations weaken the pinning effect of point disorder and in the region \( T^* < T < T' \), where \( T' \) is a temperature below which the pinning potential and \( H_{irr}(T) \) grows rapidly, the entanglement transition line is given by

\[
B_{en} \simeq B_{en}(0) \left( \frac{T^*}{T} \right)^{10/3} \exp((2c/3)(T/T^*)^3),
\]

where \( c \) is a constant of order unity. In the higher temperature region \( T > T' \), the disordering line turns into the vortex lattice melting line \( B_m \). The condition \( \langle u_{ab}^2 \rangle \simeq c_L^2 a_c^2 \) corresponds to the pure thermal melting without disorder fluctuations, so the vortex melting line \( B_m(T) \) decreases with increasing temperature. Thus the stability of the Bragg Glass in the high-\( T \) region is limited by the vortex lattice melting line.

Similar qualitative results as obtained by Ertas and Nelson [41] have been worked out with a different theoretical approach by Gianmarchi and Le Doussal [39]. The second magnetization peak line observed recently by Nishizaki et al. [49] is in good agreement with the entanglement transition predicted by these models. Additionally, the onset of the increase of the critical current density \( j_c \) with increasing field coincides approximately with the field above which the intensity of Bragg peaks
in neutron diffraction experiments suddenly decrease [38]. This gives further experimental evidence for the existence of a low-temperature, low-field Bragg glass. Nevertheless, the measured large magnitude of the increase of the critical current density \( j_c \) at the crossover, cannot be explained by any of these theories.

As we mentioned before, above the melting transition \( B_m(T) \), the vortex lattice undergoes a transformation into some liquid phase with reduced vortex pinning. Above the second peak transition \( B_{sp}(T) \), in contrast, a new phase with enhanced pinning is obtained. Two different phases at low and high temperatures must therefore exist above the entire phase transition line. One may thus anticipate the existence of another vertical phase transition or crossover line that separates the two high field phases. We will return to this subject in section 2.4.3.1, where the pinning diagram for layered superconductors proposed by Wagner et al. [12-14] is described.

### 2.3.3 Irreversibility Line

One of the widely studied features in type II superconductors is the experimentally observed irreversibility line (IL) in the phase diagram, which separates the reversible magnetization at high temperatures from the hysteretic behaviour at lower temperatures. Recent results in high temperature superconductors [3] indicate, that the irreversibility line is a result of irreversible shielding currents which are related to Bean-Livingston surface barriers [42, 43] and geometrical barriers [44]. The main characteristic of these barriers is that they inhibit the vortex entry into the superconductor, while they do not act significantly against their exit [63]. In the absence of bulk pinning, hysteresis loops are therefore obtained which are asymmetric with respect to zero magnetic moment. This is in contrast to the symmetric hysteresis loops associated with the Bean critical state and bulk pinning of vortices.

The vortex penetration through Bean-Livingston surface barriers by a thermally activated process is predicted to result in an exponential temperature dependence of the irreversibility line [64, 65]. A recent theoretical analysis of such thermal activation in highly anisotropic superconductors predicts [66]

\[
H_{IL}(T) \simeq \frac{H_{c2}T_0}{2T} \exp \left( -\frac{2T}{T_0} \right),
\]  

(2.15)
where $H_{c2} = (\Phi_0/2\pi \xi^2)$ is the upper critical field and $T_0 \simeq \epsilon_0 d/30$ with $\epsilon_0 = (\Phi_0/4\pi \lambda)^2$. This dependence agrees favorably with numerous experimentally observed behaviour of the irreversibility line for intermediate temperatures [3,67,68]. At high temperatures, geometrical barriers becomes active, and the temperature dependence of the irreversibility line in perpendicular to the $ab$-planes applied magnetic field is predicted to be [44]

$$H_{IL}(T) \simeq \frac{3}{2} H_{c1} \left(1 - \frac{T}{T_c}\right)$$

(2.16)

where $H_{c1} = (\Phi_0/4\pi \lambda^2) \ln(\lambda/\xi)$ is the lower critical field. For high temperatures this is again in good agreement with experimental results [3].

### 2.4 Pinning Diagram of Layered Type II Superconductors

The pinning diagram describes the pinning properties of the disordered vortex lattice in the mixed state and is, contrary to the phase diagram, not an equilibrium feature. The various regimes in this diagram specify the geometry of collectively pinned domains in the vortex matter, which are relevant in the determination of the critical current density $j_c$ and the activation barriers $U_c$ for vortex creep. In this section we will closely follow the theoretical approach by Wagner et al. [12-14], who generalized the calculations for the pinning diagram to layered superconductors with all degrees of anisotropy and intermediate disorder strength. Before it has only been derived for superconductors with small anisotropy [26,36,69,70].

The outline of this chapter is as follows: In section 2.4.1, different types of pinning are discussed and the disordered pinning potential $U_{pin}$ is introduced. In section 2.4.2 the concept of weak collective pinning and the associated collective pinning lengths $R_c$ and $L_c$ are presented. And finally, in section 2.4.3, the $B$-$T$ pinning diagrams calculated in [13] for strongly layered and weak anisotropic superconductors are mapped out.
2.4.1 Pinning

Quenched defects in superconducting materials produce a static disorder environment which pins the vortices. These defects are either intrinsic such as oxygen vacancies, impurities, twin or grain boundaries, or artificially induced columnar defects as for instance heavy-ion irradiation tracks. In this chapter only the intrinsic pinning due to pointlike defects perturbing the superconductor on a scale smaller than the coherence length $\xi$ is discussed, since the samples investigated in this thesis are characterized by this type of pinning.

Within the GL theory, two possible ways of quenched disorder can be thought of: First, defects weaken the superconducting state by reducing the critical temperature locally ($\delta T_c$-pinning). Second, defects reduce the mean free path $l$ of electrons through scattering; this enters the GL free energy in the form of random fluctuations of the effective electron masses $m_{ab}$ and $m_c$ ($\delta l$-pinning). Both situations can be described by means of a disordered potential $U_{pin}(r)$. In the case of a large density of uncorrelated weak point defects this random potential can be assumed to be Gaussian distributed with zero mean and short-range correlations,

$$\langle U_{pin}(r) \rangle = 0, \quad (2.17)$$

$$\langle U_{pin}(r)U_{pin}(r') \rangle = \gamma \delta^3(r - r'). \quad (2.18)$$

The parameter $\gamma$ measures the disorder strength and is supposed to be small. Further, this parameter depends on temperature and vanishes for $T \to T_c$; for $\delta T_c$-pinning, $\gamma \propto (1 - T/T_c)^2$, while for $\delta l$-pinning $\gamma \propto (1 - T/T_c)^4$ [26]. At finite temperatures the disorder potential is smeared by thermal fluctuations $\langle u^2(T) \rangle_{\text{th}}$ in the displacement and pinning is consequently weakened. For this case, using the dynamic approach, Feigel'man and Vinokur [69] showed that the pinning energy is effectively reduced by the factor $\xi/r(T)$, where $r(T) = \sqrt{\xi^2 + \langle u^2(T) \rangle_{\text{th}}}$ is the effective average distance between two minima in the energy landscape.
2.4.2 Weak Collective Pinning Theory

Larkin [71] showed that in less than four space dimensions \( D < 4 \) weak short-range correlated quenched disorder always destroys the translational long range order of the vortex lattice and breaks the system up into elastically independent and collectively pinned domains called vortex bundles of volume \( V_c = R_c^2 L_c \). The transverse collective pinning radius \( R_c \) and the longitudinal collective pinning length \( L_c \) define the boundaries of these domains with almost perfect order and are relevant for the determination of the critical current and the activation barriers for vortex creep.

In the following, the argument leading to Larkin's conclusion in the limit of zero temperature is given. Consider a region of perfect lattice with stiff vortices of volume \( V = R^2 L \). The average pinning energy \( \langle E_{\text{pin}}(R, L) \rangle \) of this region is zero because of equation (2.17). Nevertheless, the fluctuations of the pinning energy do not vanish, and the average pinning energy \( \langle E_{\text{pin}}^2(R, L) \rangle \) is given by [13]

\[
\langle E_{\text{pin}}^2(R, L) \rangle = \int_V d^3r d^3r' \langle E_{\text{pin}}(r, 0) E_{\text{pin}}(r', 0) \rangle = \pi \gamma \frac{\xi^2}{a_0^2} R^2 L. \quad (2.19)
\]

Thus, a region of straight vortices of volume \( V = R^2 L \) can typically gain the pinning energy \( E_{\text{pin}}(R, L) = \sqrt{\langle E_{\text{pin}}^2(R, L) \rangle} \), implying that the pinning force \( F_{\text{pin}} \) acting on this region also scales with \( \sqrt{V} \). On the other hand, the Lorentz force that arises when applying a current increases linearly with volume. Hence, in the presence of an external current, a region of stiff vortices is not pinned and the critical current density \( j_c \) vanishes. In reality, the vortices have a finite elasticity and can accommodate to the disorder potential. A displacement beyond the correlation length of the fluctuations in the pinning energy which is about \( \xi \) cuts off the sublinear growth of \( E_{\text{pin}}(R, L) \) at the lengths \( R_c \) and \( L_c \). Each of these regions of volume \( V_c = R_c^2 L_c \) (vortex bundles) is pinned collectively and independently. The collective pinning lengths \( R_c \) and \( L_c \) are determined by minimizing the total energy with respect to \( R \) and \( L \), where the total energy is the sum of the typical shear energy \( E_{\text{shear}} \sim c_{66} (\xi/R_c)^2 V_c \), the tilt energy \( E_{\text{tilt}} \sim c_{44} (\xi/L_c)^2 V_c \) and the pinning energy \( E_{\text{pin}}(R_c, L_c) \). Within the method of dimensional estimates the critical current density \( j_c \) is obtained by equating the pinning force and the Lorentz force which is proportional to the applied current.
2.4.3 Pinning Regimes of the Vortex Lattice

In the previous section, the so called 3D vortex bundle pinning regime has been described, where the three relevant energy scales match, \( E_{\text{shear}} \approx E_{\text{tilt}} \approx E_{\text{pin}} \). However, at low inductions the elastic shear modulus vanishes rapidly and the shear energy becomes smaller than the other two energies, \( E_{\text{shear}} < E_{\text{tilt}} \approx E_{\text{pin}} \). As a consequence, \( R_c \) drops below the lattice spacing \( a_0 \) and the collectively pinned objects are now vortex line segments of length \( L_c \). This regime is accordingly called 1D single vortex pinning regime. In the case of continuous anisotropic superconductors we thus have two different pinning regions: a vortex bundle regime at high and a single vortex regime at low inductions.

In discrete layered superconductors one must further take into account the discrete structure of the material in the c-direction. In section 2.2.2 it was argued that with the magnetic field perpendicular to the superconducting layers the vortex lines can break up into weakly coupled pancake vortices. This opens the possibility for two (strong) pinning regimes in addition to the 1D and 3D (weak) pinning regimes: At low fields, the absence of interaction between the pancake vortices allows for their free accommodation to the pinning potential, thus leading to strong pinning of individual pancake vortices. This regime is named 0D single-pancake-vortex pinning regime and is characterized by \( E_{\text{shear}}; E_{\text{tilt}} < E_{\text{pin}} \) or in terms of collective pinning length \( R_c = a_0, L_c = d \). With increasing magnetic field the shear energy increases and a state where the pancake vortices of one layer are pinned collectively, but independently of those of other layers \( E_{\text{tilt}} < E_{\text{shear}} \approx E_{\text{pin}} \), i.e., \( R_c > a_0, L_c = d \) is expected. This regime 2D is accordingly called pancake-vortex-bundle regime.

The boundaries and properties of these four pinning regimes have been theoretically investigated for superconducting layered materials for the case where the magnetic induction is perpendicular to the planes by Wagner et al. [12-14]. Using collective pinning theory and introducing the new concept of variable range thermal smoothing of the pinning potential, they map out the pinning diagram and find the regimes of single-pancake-vortex (0D), single-vortex (1D), pancake-vortex-bundle (2D) and vortex-bundle (3D) pinning in the \( B-T \) diagram as a function of
the strength of the Josephson coupling between the layers and the strength of the disorder potential. In the following sections we will present their results for discrete layered superconductors and for continuous anisotropic superconductors.

It is important to mention that the results of [12-14] are restricted to the regime where one can rely on a well-defined elastic free energy. This regime is limited by the melting of the vortex lattice as studied in section 2.3.1. Hence, large portions of the diagrams in figure 2.3 and 2.4, especially for high fields and temperatures, are unphysical - the exact boundaries depend strongly on material parameters and have to be determined in each specific case.

2.4.3.1 Discrete Layered Superconductors

The $B$-$T$ pinning diagram for a layered superconductor with decoupled layers (anisotropy parameter $\epsilon = 0$) as proposed by Wagner et al. [13,14] is illustrated in figure 2.3. The low induction part $B < B_\lambda \simeq (\Phi_0/\lambda^2) (\simeq 10^3 \text{ G for BSCCO})$ of this pinning diagram is weakly modified considering in addition Josephson coupling between superconducting layers (anisotropy parameter $\epsilon \ll 1$) [13,14].

The strong pancake vortex pinning region at low temperatures $T < T_{pc}$ ($T_{pc}$ is defined as pancake depinning temperature) is divided into two parts: At $T < T_g$ thermal effects are irrelevant and the pancakes vortices are strongly pinned individually [(0-0)D regime], while at $T_g < T < T_{pc}$ thermal smoothing of the pinning potential takes place, and the pancake vortices are weak elastically coupled between the layers [(0-1)D and (0-3)D regimes]. Accordingly, the dashed thick line at the temperature of glassy response $T_g$ indicates a jump in the collective pinning length $L_c$ and in the critical current density $j_c$. For temperatures $T > T_{pc}$ and low inductions $B$, vortex segments of length $L_c > \lambda$ are pinned collectively (1D regime). Further, for $T > T_{dp}^s$ ($T_{dp}^s$ is defined as single vortex depinning temperature) the interaction among the vortices is relevant and short range lattice order persists over several lattice constants. The remaining two areas show the pancake-vortex-bundle [2D] and the vortex-bundle [3D] pinning regime.

In table 2.1 the different temperatures and the different magnetic inductions quoted above and in figure 2.3 are expressed in terms of the line energy $\epsilon_o$, the pancake
vortex energy \( E_o \), the electromagnetic elastic energy per pancake vortex \( E_{em} \), the Josephson elastic energy per pancake vortex \( E_J \), the electromagnetic-Josephson elastic energy per pancake vortex \( E_{em,J} \), and the disorder parameter \( U_p \).
\[
T_g \simeq U_p \left[ \ln \left( \frac{U_p}{E_{em,j}} \right) \right]^{-1/2} \\
T_p \simeq U_p \\
T_{pc} \simeq U_p \left[ \ln \left( \frac{U_p}{E_{em,j}} \right) \right]^{1/2} \\
T_{pd}^a \simeq \left( \frac{E_p U_p^2}{\mu} \right)^{1/3} \\
B_{que} \simeq B_\lambda \left( \ln \left( \frac{1}{\xi^2} \right) \right)^{2} \\
B_\lambda = \frac{\Phi_0}{\lambda^2} \\
B_\Lambda = \frac{\Phi_0}{\Lambda^2} \\
B_{00} \simeq B_\lambda \ln \left( \frac{\Lambda^2}{\xi^2} \right) \left( \frac{E_J}{U_p} \right) \\
B_{02} \simeq B_\lambda \left( \frac{U_p}{E_{em}} \right) \\
B_{23} \simeq B_\lambda \left( \frac{U_p}{E_J} \right)^{4/3}
\]

Table 2.1: Temperatures and magnetic inductions expressed in terms of the line
energy \( \epsilon_o = (\Phi_0/4\pi \lambda)^2 \), the elastic energy per pancake due to electromagnetic in-
teractions \( E_{em} = \epsilon_o d (\xi/\lambda)^2 \), the modified pancake vortex energy \( E_o = \epsilon_o d (\xi/d)^2 \),
the Josephson elastic energy per pancake vortex \( E_J = \epsilon_o d (\xi/d)^2 \), the
electromagnetic-Josephson elastic energy per pancake \( E_{em,j} = \epsilon_o d (\xi^2/\lambda \Lambda) \), and
the disorder parameter \( U_p = \sqrt{\gamma \xi^2 d} \). The parameter \( U_p \) can be estimated from
experiments measuring the critical current density at low \( T \) and \( B \): \( j_c/j_o \sim U_p/\epsilon_o d \).
The parameters \( \lambda \) and \( \xi \) denote the planar London penetration depth resp. coherence
length, \( \Lambda = d/\epsilon \) the Josephson screening length, \( d \) the interlayer spacing, \( \epsilon \)
the anisotropy parameter, \( \Phi_0 = hc/2e \) the flux unit, and \( \gamma \) the disorder parameter.

For low magnetic inductions \( B \), the crossover between the regime of individu-
ally strongly pinned pancake vortices (0-0)D and the regime of weak elastically
coupled pancake vortices (0-1)D and (0-3)D, is temperature independent. Also the
crossover between the pinning regions (0-1)D and (0-3)D and the single-vortex (1D)
resp. the vortex-bundle (3D) weak pinning regimes, are predicted to be temperature
independent. Instead, the transition from the single-vortex pinning regime (1D) to
vortex-bundle pinning regime (3D) which falls in the low field regime investigated
in this thesis, is temperature dependent and is given by
2. Vortex Properties in Layered Type II Superconductors

\[
\frac{B_{13}(T)}{B_\lambda} = \begin{cases} 
\left( \frac{2T}{3T_g} + \frac{T}{T_g} + \frac{2}{3} \ln\left( \frac{1}{\epsilon} \right) \right)^{-2} & T_g \ln(\frac{\lambda T}{d}) < T < T_{pc} \\
\left[ \ln\left( \frac{1}{\epsilon} \frac{T^3}{E_j U_p^2} \right) \right]^{2/3} & T_{pc} < T < T_{dp}^s \\
\left( \ln\left( \frac{1}{\epsilon^2} \right) \right)^{-2} \left( \frac{T_{dp}^s}{T} \right)^6 & T_{dp}^s < T
\end{cases}
\] (2.20)

For the temperature dependence of the various crossovers at high magnetic inductions, such as the temperature dependence of the pinning radius \( R_c \), the pinning length \( L_c \), the pinning energy \( U_c \) and the critical current density \( j_c \) in the different regimes, we refer to [12–14].

2.4.3.2 Continuous Anisotropic Superconductors

The pinning diagram for continuous anisotropic superconductors is shown in figure 2.4. The strong pinning regimes (0D and 2D) disappear, leaving only the single-vortex (1D) and the vortex-bundle (3D) weak pinning regimes. The fat dotted line at \( T \approx T_{dp}^j \left( \ln(U_p/E_{wp}) \right)^{1/3} \) marks the position of a jump in the pinning length \( L_c \) from \( \epsilon \lambda \) to a value beyond \( \lambda \). \( T_{dp}^j \) is defined as the depinning temperature of single vortices in continuous anisotropic superconductors. As the anisotropy parameter \( \epsilon \) is increased, \( T_{dp}^j \) approaches \( T_{dp}^s \) and the discontinuity at \( T_{dp}^j \left( \ln(U_p/E_{wp}) \right)^{1/3} \) eventually disappears for \( \epsilon \to 1 \).

The crossover line \( B_{13}(T) \) between the 1D and the 3D pinning regime is for \( T \ll T_{dp}^j \), temperature independent and equal to the small-bundle crossover field \( B_{sb} \). The dashed line within the 3D pinning regime slightly above \( B_{13}(T) \) at low temperatures marks the position of the large-bundle boundary \( B_{1b}(T) \) below which the pinning volume changes exponentially (small-bundle regime). Above the dotted line which goes through the 3D regime at \( T > T_{dp}^j \) the characteristics are determined by the Josephson coupling, whereas below the electromagnetic expressions hold.

In table 2.2 the temperatures and the magnetic inductions quoted above and in figure 2.4 are expressed in terms of the disorder parameter \( U_p \), the line energy \( \epsilon_o \), and the Josephson elastic energy per pancake vortex \( E_J \).
Figure 2.4: $b-t$-pinning diagram of a layered superconductor with large anisotropy parameter $\epsilon > \epsilon_{\text{an}} = (d\lambda)\sqrt{U_p/E_{\text{em}}}$ (or equivalently $U_p < E_d$) as presented in [13, 14] (logarithmic scales, $t = T/T_c$, $b = B/H_{c2}$; parameters similar to BSCCO: $\lambda = 1500 \text{Å}, \xi = 25 \text{Å}, d = 15 \text{Å}, T_c = 90 \text{K}, H_{c2} = 50 \text{T}$). Results are shown for $\epsilon = 1/10$ and moderate pinning $U_p = 4 \text{K}$.
Table 2.2: Temperatures and magnetic inductions expressed in terms of the disorder parameter $U_p = \sqrt{\gamma \xi d}$, the line energy $\varepsilon_0 = (\Phi_0/4\pi \lambda)^2$, and the Josephson elastic energy per pancake vortex $E_J = \varepsilon_0 d (\xi/d)^2$. The parameters $\lambda$ and $\xi$ denote the planar London penetration depth resp. coherence length, $d$ the interlayer spacing, $\Phi_0 = \hbar c/2e$ the flux unit, and $\gamma$ the disorder parameter.

For the temperature dependence of the crossover field $B_{13}(T)$ and of the pinning radius $R_c$, the pinning length $L_c$, the pinning energy $U_c$ and the critical current density $j_c$ in the different regimes, we refer to [13,14].
3 Experimental Arrangement

After the discovery of the high temperature superconductors (HTSC) two magnetometers with an applicability range of temperatures between 4 K and 200 K have been built in our laboratory. A first version of such a “high temperature rf-SQUID magnetometer” has been successfully constructed during his Ph. D. Thesis by T. Teruzzi [72], following the design of Prof. Dr. A.C. Mota. Shortly after, motivated by the continuous and strong interest for the HTSCs, a second version of the above mentioned magnetometer equipped with a dc-SQUID has been built during his Ph. D. Thesis by M. Nideröst [73].

One of the main advantages of SQUID (Superconducting Quantum Interference Device) magnetometers with respect to other magnetometers, is their high sensitivity. In our case we obtain a measuring sensitivity at both type of our magnetometers of about $10^{-2} \Phi_0$ which corresponds to 1-2 $\Phi_0$ at the sample. A further important characteristic of our SQUID magnetometers is that the sample remains stationary in the pick-up coil during the measurements. Commercial SQUID magnetometers usually adopt a data acquisition technique which consists in cycling the position of the sample up and down through a pick-up coil in a typical time scale of one minute and subsequently integrating over the resulting signal. In the case of fast signal variations, such cycling procedures lead to large systematic errors which can be avoided by keeping the sample stationary.

Since the cryostat and the measuring system have been described in detail in references [72,73], only an overview of our experimental arrangements is given here. This short description of the two magnetometers is followed by a description of the
experimental procedures. At the end of this chapter, all the samples investigated within this work are characterized.

3.1 Measuring System

The rf-SQUID magnetometer and the dc-SQUID magnetometer differ from each other essentially by the experimental cell and the SQUID sensors and electronics. The experimental arrangement for temperature regulation and control are very similar in both cases. In the next section we will give a short overview of this temperature regulation and control of the magnetometers while in the subsequent sections a short outline of the main characteristics of the two systems are presented.

3.1.1 Temperature Regulation and Control

In figure 3.1 a schematic overview of the experimental apparatus for temperature regulation and control used in both magnetometers is shown.

The magnetometers are mounted inside a superinsulated dewar, containing about 20 liters of liquid 4He. Suspended from the brass top-plate into the cryogenic environment is the vacuum can and SQUID sensor. The lower end of the vacuum can, which is the experimental cell, is made up of Stycast 1266. A special seal was constructed to attach this Epoxy part to the stainless steel of the vacuum can. The sample in the experimental cell can be put in thermal contact with the external liquid 4He-bath by means of the 4He exchange gas in the vacuum can. The sample is placed in the experimental cell by a top loading procedure which does not require any warming up of the cryostat to room temperature.

In other to reduce the earth’s magnetic field in the experimental space, a μ-metal shield, consisting of a bottom closed cylinder of cryoperm [74], has been placed around the cell, at the bottom of the dewar. The residual field in the detection region has been measured to be less than 10 mOe.

The SQUID sensor is directly immersed in the liquid helium bath and is located next to the experimental cell, at the lowest end of a SQUID probe. This configuration
Figure 3.1: Schematic overview of the experimental apparatus of the two magnetometers.
guarantees good thermal stability. The sensor is coupled to the pick-up coil by shielded niobium-titanium leads immersed in the helium bath.

The temperature regulation in both cryostats is mainly controlled by two heat flows: Heat is transferred from the room temperature environment through the top loading insert down to the sample. On the other hand, heat is carried away from the insert and the sample to the helium bath through a $^4$He-exchange gas in the vacuum chamber. By regulating the pressure of this gas, the equilibrium temperature at the sample can be chosen between 4.2 K and approximately 100 K. An additional electrical manganese heater connected to an electronic temperature controller is used for fine regulation and stabilization of the temperature as well as for obtaining temperatures above 100 K. For the temperature measurements a Si-diode thermometer in the rf-SQUID and a GaAlAs-diode thermometer in the dc-SQUID magnetometer have been chosen.

3.1.2 *rf*-SQUID Based Magnetometry

A cross-section of the experimental cell of the *rf*-SQUID based magnetometer, which is an extension of the vacuum-can, is displayed in figure 3.2. In order to minimize the presence of magnetic impurities and eddy currents in the detection region, the main parts of the cell were made of Stycast 1266 [75], which is a non-magnetic and non conducting epoxy resin [76].

The coil arrangement consists of three cylindrical coils wound around the external part of the experimental cell. With this arrangement *ac*-susceptibility as well as *dc*-magnetization measurements can be performed. All three coils are made of niobium-titanium wire.

The inner pick-up coil, a first order gradiometer, is a pair of five to five windings wound in opposite direction on the stycast extension of the vacuum can. In an ideal geometry, a first order gradiometer does not respond to homogeneous magnetic fields, so that any response induced in the pick up coil is only due to magnetic flux changes inside a sample placed in the center of one of the loops. Signals coming from non-ideal features of the gradiometer are measured by running the cryostat with an empty sample holder (background). The first order gradiometer is connected by
3.1 Measuring System

Figure 3.2: The experimental cell of the rf-SQUID magnetometer.
3. Experimental Arrangement

twisted and shielded NbTi wires to a signal coil which is inductively coupled to the SQUID sensor. The signal coil and the SQUID sensor are placed inside the SQUID sensor housing (see figure 3.1). The current induced in the gradiometer by a flux change in the sample induces via the signal coil a flux change at the SQUID sensor. The SQUID control unit transforms this input current into a dc output voltage which is directly proportional to the flux change at the sample. A digital flux counter (DFC) offers the possibility of converting this dc output voltage in units of the flux quantum $\Phi_0$. The flux change measured at the SQUID sensor is proportional to the flux change at the sample.

The ac-field coil is wound on top of the gradiometer coils so that the axial ac-field inhomogeneity respect to the pick up coil is less than 0.1 % [72]. ac-susceptibility measurements are performed by exciting the ac-field coil by a current generated by the RBU mutual inductance bridge [77]. It can operate at four different frequencies: 16, 32, 80 and 160 Hz. For the amplitude of the excitation field, nine values between $H_{ac} = 0.15$ mOe and $H_{ac} = 76$ mOe can be chosen. The oscillating field induces a current in the flux transformer which depends on the mutual inductance between the ac-field coil and the gradiometer. The mutual inductance is influenced by the magnetic susceptibility of the sample. The induced current is amplified by the SQUID unit and its output voltage is further amplified by the two phase lock-in analyzer. The RBU bridge balances the induced current to zero through a mutual inductance coil situated between the gradiometer and the signal coil, thus the values of the in phase $\chi'$ and out of phase component $\chi''$ of the sample’s complex magnetic susceptibility can be determined.

The dc-field coil is wound on a support screwed around the lower part of the vacuum can. Its axial field inhomogeneity at the position of the gradiometer is less than 0.05 % [72]. This coil is characterized by a field to current ratio of $1800 \pm 50$ Oe/A. Two different custom made constant current sources, which can supply maximal currents of 100 mA and 500 mA respectively, are used to energize the field coil. Their main common characteristics is an extremely low noise level. This is an essential condition when measuring with SQUID sensors while the coil is operating in the non-persistent mode.
3.1.3 dc-SQUID Based Magnetometry

A cross-section of the experimental cell of the dc-SQUID based magnetometer, is displayed in figure 3.3. In order to avoid eddy currents, the experimental cell is entirely built out of Stycast 1266 [75], as in the case of the rf-SQUID based magnetometer. The coil arrangement consists of two cylindrical coils wound around the external part of the experimental cell. With this arrangement only dc-magnetization measurements can be performed. The two coils are made of niobium-titanium wire.

The inner coil is the pick up coil, wound as a three to six to three second order gradiometer. This gradiometer is inductively coupled to a dc-SQUID acting as a low-noise current to voltage transformer and amplifier. dc-SQUID sensors and their electronics have some important advantages as compared to rf-SQUIDs: apart from the lower input noise level, they exhibit a one order of magnitude higher slew rate, a larger dynamic range, and a much faster reset time. Due to these qualities, dc-SQUIDs are ideal for ultra sensitive recording of very fast signal variations.

The dc-magnetic field $H$ is generated by a superconducting coil wound on a support concentrically embedding the gradiometer. The coil is characterized by a field to current ratio of $5000 \pm 50 \text{ Oe/A}$. The relatively large coil length of 10 cm guarantees a homogeneous field distribution in the gradiometer region [78]. Furthermore, the coil has been covered with a layer of manganin wire acting as a heater, thermally insulated from the He-bath by 2-3 layers of mylar foil. This configuration allows to heat the NbTi-coil within the He-bath above its critical temperature $T_c \approx 9.2 \text{ K}$ in order to remove any flux which has been trapped when the field $H$ was cycled above the lower critical field $H_{c1} (\approx 150 \text{ Oe})$ of the NbTi wires. Thus one can avoid disturbances at the sample position by flux creep in the field coil. In addition to the two custom made constant current sources discussed in the previous section, also a third one which can supply a maximal current of 1 A, is used to energize the dc-field coil.
Figure 3.3: The experimental cell of the dc-SQUID magnetometer.
3.2 Experimental Procedure

The two magnetometers described in the previous section allow to perform various types of measurements. However, all measurements presented in this work can be grouped into two classes: isothermal and non-isothermal measurements. The isothermal measurements consist of $ac$-susceptibility and $dc$-magnetization field sweeps and time relaxation measurements of the remanent magnetization $M_{\text{rem}}$ while the non-isothermal measurements are $ac$-susceptibility and $dc$-magnetization temperature sweeps.

Since only the $rf$-SQUID magnetometer allows to perform $ac$-susceptibility measurements, most of the data presented in this work have been measured on this magnetometer. However, due to fact that the $dc$-SQUID can follow signal changes that are one order of magnitude faster than the $rf$-SQUID, the time relaxation measurements of the remanent magnetization $M_{\text{rem}}$ have been performed on the $dc$-SQUID magnetometer.

All the reported measurements have been repeated either during an empty run (no specimen in the magnetometer) or above the critical temperature $T_c$ of the sample, following exactly the same procedure as for the measurements of the sample in the superconducting state. Subsequently, these background signals have been subtracted from the raw data in order to obtain the real sample signal.

3.2.1 Isothermal Measurements

For the isothermal measuring procedures, the sample is first zero field cooled (ZFC) (residual field of the cryostat $H_{\text{rem}} \lesssim 10 \text{ mOe}$) starting from temperatures well above $T_c$ and then stabilized at a desired temperature $T$. This guarantees an almost completely magnetization free initial state of the sample and defines the starting point of the experiment in the $H$-$T$ diagram.
ac-Susceptibility and dc-Magnetization Field Sweeps

For the measurement of the ac-susceptibility or the dc-magnetization curves the external field \( H \) is cycled from zero up to the maximal value \( H_{\text{max}} \) and back to zero again. During this time the complex magnetic susceptibility \( \chi(H) \) or the magnetization \( M(H) \) is recorded, respectively. The superconducting field coil works in a non-persistent mode and is energized by a low-noise power supply. The external magnetic field \( H \) is swept stepwise or continuously for average cycling rates \( \frac{dH}{dt} \) between \( 10^{-5} \) and \( 10^{-1} \) Oe/s. However, the cycling rate has to be chosen such that during the measurements the rate of the flux change at the SQUID sensor never exceeds the slew rate of the SQUID itself or of its electronics. Once the external field is back to zero the sample is heated above its critical temperature \( T_c \) in order to remove the trapped flux. At that point, the sample is again magnetization free and a further measurement can be started at a different temperature.

Decay of the Magnetization

Whenever the externally applied magnetic field \( H \) is cycled from zero up to the maximal value \( H_{\text{max}} > H_p \) and back to zero again a metastable remanent magnetization \( M_{\text{rem}} \) is left in irreversible type II superconductors. The present measurements deal with the relaxation of \( M_{\text{rem}} \) as a function of time.

In order to record the time relaxation of the remanent magnetization \( M_{\text{rem}} \) starting from a configuration where the flux density gradient is close to the critical state, a new experimental procedure has been developed within the Ph. D. Thesis of M. Nideröst on the dc-SQUID magnetometer [73, 78, 79]. After stabilizing the temperature \( T \), the magnetic field \( H \) is gradually increased from zero to \( H_{\text{max}} \) before being linearly removed at a rate of 9 T/s. This fast rate is achieved by shorting the superconducting coil (\( L \approx 7 \) H) over a voltage dependent resistor [80].

As a time origin, the moment at which the magnetic field \( H \) starts to be removed is chosen. For this measuring procedure the uncertainty for the time origin of the process of vortex creep is smaller than \( 1.8 \cdot 10^{-2} \) s. The initial behaviour of the relaxation data is therefore well defined, since it is possible to follow the behaviour of the current density \( j(t) \) starting from values close to the critical current density \( j_c \). After measuring the relaxation of the remanent magnetization \( M_{\text{rem}} \) for about seven
3.2 Experimental Procedure

Time decades ($10^{-2}$ s → $10^{+5}$ s), the sample is heated above $T_c$ in order to record its residual magnetization $M_{\text{res}}$. The total $M_{\text{rem}}$ is then given by the sum of the relaxed part of $M_{\text{rem}}$ and $M_{\text{res}}$.

3.2.2 Non-Isothermal Measurements

The temperature controllers of the two magnetometers allow to sweep the temperature of the sample monotonically with any desired rate $dT/dt$ over the whole temperature interval $4 \text{K} \leq T \leq 200 \text{K}$. This offers the possibility of measuring the temperature dependence of the sample's complex susceptibility $\chi(T)$ or magnetization $M(T)$ with or without applied magnetic field.

For zero field cooling (ZFC) experiments the sample is first cooled in the residual field of the cryostat from a temperature well above $T_c$ down to a temperature $T > 4 \text{K}$. Afterwards, a magnetic field $H$ is applied and the susceptibility $\chi(T) = \chi'(T) + i\chi''(T)$ or the magnetization $M(T)$ is recorded while the sample is heated above $T_c$. By further cooling the specimen below $T_c$, without removing the applied magnetic field, a field cooling (FC) measurement can be performed. If the sample is heated again above $T_c$ without the applied magnetic field, the remanent magnetization $M_{\text{rem}}$ is determined.
3.3 The Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ Compound

Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ (BSCCO) is one of the most investigated high temperature superconductors. HTSCs differ from low temperature superconductors in a number of properties. Apart from the high mean-field transition temperature $T_c$, they have large magnetic penetration depths $\lambda$, small coherence length $\xi$, and a layered, quasi two dimensional crystal structure. The crystal structure of BSCCO is shown in figure 3.4.

The unit cell of the Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ compound has an orthorhombic crystal structure with lattice parameters $a = 3.8\,\text{Å}$, $b = 3.8\,\text{Å}$, and $c = 30.8\,\text{Å}$ [81, 82]. The compound has a strongly layered structure: sets of CuO$_2$-double layers are separated by a SrO, two BiO and another SrO layer. This layered structure leads to a large anisotropy between the normal state resistivity $\rho_{ab}$ measured in the ab-planes, which decreases linearly with temperature, and the resistivity $\rho_c (\sim 10^5 \rho_{ab})$ measured along the c-axis, which show semiconducting properties [83]. Thus there is a huge anisotropy of the charge carrier effective masses, characterized by the anisotropy parameter $\varepsilon = \sqrt{m_{ab}/m_{c}}$. The behaviour of $\rho_{ab}$ is associated with the metallicity of the CuO$_2$ layers, whereas the behaviour of $\rho_c$ is explained by the insulating character of the BiO layers, which together with the SrO layer acts as an electric charge reservoir.

The charge carrier effective mass anisotropy is reflected in the anisotropy of superconducting parameters $\lambda$ and $\xi$. The values of the Ginzburg-Landau anisotropy parameter $1/\varepsilon = \lambda_c/\lambda_{ab} = \xi_{ab}/\xi_c$ obtained from magnetic torque [84], muon spin rotation [85] and microwave dissipation measurements [86] for the Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ compound lie in between 50 and 350. The zero temperature extrapolation of the in-plane penetration depth $\lambda_{ab}(0)$ reported in the literature [85, 87–89], is $\lambda_{ab}(0) \approx 1800 - 3000\,\text{Å}$. The relatively large spectrum of values for the anisotropy parameter $1/\varepsilon$ and the penetration depth $\lambda$ is related to the fact, that these values depend on the oxygen stoichiometry of the investigated single crystal sample [88,90]. Values of the coherence length in the ab-direction $\xi_{ab}(0)$ have been determined from the upper critical field in c-direction $H_{c2}(T = 0)$ estimated between 75 T and 230 T [1, 91]. This leads to a coherence length $\xi_{ab}(0)$ of $12 - 20\,\text{Å}$. Apart
from temperatures very close to the critical temperature $T_c$, the coherence length in the $c$-direction $\xi_c \approx \sqrt{\xi_{ab}}$ is therefore much smaller than the interlayer distance $d = 15.4 \ \text{Å}$ [81, 82]. Consequently, in a large part of the mixed state phase diagram, superconducting properties are then better described by the Lawrence-Doniach model for Josephson coupled superconducting layers (see section 2.2.2) than by the anisotropic Ginzburg-Landau theory (see section 2.2.1).
Figure 3.5: Temperature dependence of the ac-susceptibility. The measurement has been taken using an ac-amplitude of 7.7 mOe ($H_{ac} \perp ab$) and a frequency of 160 Hz. The width of the transition, as defined by the 10% – 90% criterion, is $\Delta T_c = 1.7 \, K$.

The size of the Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ single crystal investigated in this work is $1.0 \times 1.6 \times 0.05 \, mm^3$ with the shortest dimension along the $c$-axis. The cross section is approximately rectangular. This sample has been prepared by Li et al. [81,88] with the traveling solvent floating zone method (TSFZ). The 'optimally-doped' specimen was grown in 200 kPa oxygen pressure which leads to maximal values of the transition temperature $T_c$. As obtained from zero field cooled susceptibility measurement and shown in figure 3.5, the onset of $T_c$ for the configuration with $H \perp ab$ is 93 K. The rather sharp superconducting transition $\Delta T_c = 1.7 \, K$ is an indication of the high quality of this single crystal. Further evidence of the good crystal quality comes from magneto-optical measurements performed at the École Polytechnique in Palaiseau (France) [92]. These measurements show a homogeneous flux penetration throughout the entire crystal when increasing the magnetic field above the Meissner state.
3.4 The 2H-NbSe₂ Compound

The continuous anisotropic and conventional superconductor niobium diseleneide 2H-NbSe₂ has attracted a great deal of attention over the last few years because many of its properties lie in between those of typical low-Tc superconductors and the high-Tc cuprates.

The NbSe₂ compound has a layered structure in which each Nb-atom has as nearest neighbours 6 Se-atoms arranged at the apices of a trigonal prism [93]. The crystal structure of the two-layer hexagonal form (2H-) niobium diselenide is characterized by a sequence of Nb layers which are separated by two hexagonal ordered Se layers (see figure 3.6). Within the layer the bonding is quite strong while between the layers the bonding is weak. The distance between the layers is $d = 6 \text{ Å}$ [94].

The values of the Ginzburg-Landau anisotropy parameter $1/\varepsilon = \lambda_c/\lambda_{ab} = \xi_{ab}/\xi_c$ reported in the literature from measurements of the angular dependence of the upper critical field $H_{c2}(T = 0)$ lie in between 2 and 6 [30,94-97]. Furthermore, the zero temperature values of the in-plane penetration depth $\lambda_{ab}$ reported in the literature [30,95,98], is $\lambda_{ab}(0) \approx 700 - 1300 \text{ Å}$. Values of the coherence length in the $ab$-direction $\xi_{ab}(0)$ have been determined from the upper critical field in $c$-direction $H_{c2}(T = 0)$ estimated between 3 and 5 T [94-97]. This leads to a coherence length in the $ab$-direction $\xi_{ab}$ lying in between 77 - 95 Å. The coherence length in the $c$-direction $\xi_c = \varepsilon \xi_{ab} \approx 15 - 32 \text{ Å}$ is therefore considerably larger than the distance $d = 6 \text{ Å}$ between the tri-layers Se-Nb-Se [94]. Accordingly, this compound is well described by the anisotropic Ginzburg-Landau treatment (see section 2.2.1.1).

Some of the characteristics of 2H-NbSe₂ compared to high-$T_c$ and low-$T_c$ superconductors are as follows:

Like low-$T_c$ alloy superconductors, its resistivity in the normal state is low and the low value of the dimensionless quantum resistance $Qu = e^2\rho_n/hc\xi \sim 10^{-3}$ suggests that the quantum effects relating to the flux-line lattice are unimportant.

However, as indicated by its relatively large Ginzburg number $Gi = (k_B T_c/\sqrt{2}H_c^2c\xi^3)^2 \sim 10^{-4}$ compared to that of conventional superconductors ($Gi \sim 10^{-9}$), the superconducting fluctuation effects in 2H-NbSe₂ are
expected to be more important than those in low-$T_c$ intermetallic superconductors. A feature, which distinguishes the usually prepared single crystal sample $2H$-$NbSe_2$ from both the low-$T_c$ alloy superconductors and the high-$T_c$ cuprates, is the unusually small value of the ratio of the critical current density $j_c$ to the theoretical depairing current density $j_0$ in this system. In single crystal samples of $2H$-$NbSe_2$, this ratio is of order $10^{-5}$ to $10^{-6}$ and it is perhaps the lowest amongst all low-$T_c$ alloy superconductors. Furthermore this ratio is four orders of magnitude smaller than the one observed in the purest of single crystals of high-$T_c$ cuprates. Such a low value of $j_c/j_0$ reflects the extremely weak pinning of the flux lines in $2H$-$NbSe_2$. This weakness of the pinning force has been the subject of much recent work on $2H$-$NbSe_2$. Many of these studies have utilized the Larkin-Ovchinikov [47] collective pinning theory to understand various observations related to interesting pinning characteristics (peak effect) near $T_c(H)$ [16, 99].

In this work we investigated two different $2H$-$NbSe_2$ single crystal samples. Both samples have been provided by Dr. A. Waszczak from Bell Laboratories and investigated in the group of Prof. P. H. Kes [19]. They obtained a critical temperature of $T_c = 7.3$ K [19, 20, 100]. Unfortunately, we do not have the same high precision
3.4 The $2H$-$\text{NbSe}_2$ Compound

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Figure 3.7: Temperature dependence of the ac-susceptibility of specimen I and specimen II. The measurements have been taken using an ac-amplitude of 15 mOe ($H_{ac} \perp ab$) and a frequency of 160 Hz. The width of the transition, as defined by the 10% - 90% criterion, is for the specimen I $\Delta T/T_c = 0.004$ and for the specimen II $\Delta T/T_c = 0.007$.

in the absolute value of temperature on account of small thermal gradients of the order of a tenth of a Kelvin which were difficult to account on this temperature. However, this fact does not influence the present studies.

Most of the measurements were performed on 'specimen I'. The size of this single crystal is $3.5 \times 2.5 \times 0.2 \text{ mm}^3$ with the shortest dimension parallel to the c-axis. The cross section is approximately rectangular. The sharp superconducting transition $\Delta T/T_c = 4.10^{-3}$ (see figure 3.7) is an indication of the high quality of the sample. To further reproduce the results of 'specimen I', a smaller $2H$-$\text{NbSe}_2$ single crystal has been investigated. The size of this 'specimen II' is $2.9 \times 2.2 \times 0.2 \text{ mm}^3$ with the shortest dimension parallel to the c-axis. The cross section is also approximately rectangular. As obtained from zero field cooled susceptibility measurement shown in figure 3.7, the superconducting transition is not as sharp as in 'specimen I'. However, the small width of this transition $\Delta T/T_c = 7.10^{-3} \text{ K}$ allowed us to reproduce the results obtained slightly below $T_c(H)$ by the larger sample.
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4 Low Magnetic Field Diagram of Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$

4.1 Introduction

The large mixed state regime of high temperature superconductors (HTSC) is characterized by a complicated $H$-$T$ diagram which is still a matter of controversy. Experimental investigations in the last decade have shown that this vortex phase diagram is mainly composed of three distinct regions: a rather ordered vortex lattice at low fields, a highly disordered vortex solid at high fields, and a vortex fluid at high temperatures [11,101]. However, the exact nature of the vortex structure in these several different vortex phases is actually not fully known and there is no general consensus about the type of transition between the different vortex regions. Moreover, recent Hall-probes measurements demonstrated that this $H$-$T$ diagram is even more complex and apparently displays additional new vortex phases [9]. Accordingly, the very complicate phase diagram of the high temperature superconductors is far from being completely elucidated and is thus still subject of substantial theoretical and experimental efforts.

Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ is the paramount example of high temperature superconductor showing the relevant characteristics associated with the properties of HTSC's: high anisotropy ($\epsilon \sim 10^{-2}$), high critical temperature ($T_c \sim 90$ K), and weak pinning ($j_c/j_0 \sim 10^{-2}$). The main focus of this work was to investigate the whole low field $H$-$T$ diagram of this compound using an ultra sensitive non commercial rf-SQUID-magnetometer and a high quality single crystal. For the investigations
of the vortex behaviour, susceptibility and magnetization measurements with isothermal field sweeps as well as with temperature sweeps at different constant fields have been performed. The ac- and the dc-fields have been applied perpendicular to the ab-planes of the single crystal.

In the first two sections the susceptibility measurements $\chi(T)$ and $\chi(H)$ as well as the magnetization measurements $M(T)$ and $M(H)$ are described and discussed. In section 4.4 the low field vortex matter diagram obtained from our data is presented and compared with theoretical models describing the respective phase and pinning transitions.

### 4.2 AC-Susceptibility Measurements

The data presented in this section are obtained by the response of the sample to a small amplitude ac-field superimposed on a background dc-field. Measuring the change of inductance of a pickup coil surrounding the specimen, the in-phase component $\chi'$ and the out-of-phase component $\chi''$ of the susceptibility has been determined. The in-phase component is related to the shielding capability of the currents in the sample and the out-of-phase component $\chi''$ is related to the dissipation induced by the ac-currents in the specimen. In Appendix A, three types of models giving a more detailed physical description of $\chi'$ and $\chi''$ are discussed. Among this three models, the "diffusive model" is the only one appropriate to describe superconductors [102] and at the same time independent of the amplitude $H_{ac}$ of the ac-field (linear response). As our susceptibility measurements performed on the Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ single crystal did not show any dependence on the $H_{ac}$ amplitude (see Appendix A), the data presented in this section will be discussed within this "diffusive model" [26,103].

The susceptibility measurements shown in this section have been performed with an ac-field of frequency $\omega_{ac} = 160$ Hz and an amplitude of $H_{ac} = 30$ mOe. In section 4.2.1 we will present the temperature sweep measurements $\chi(T)$ while in section 4.2.2 the field sweep measurements $\chi(H)$ are given.
4.2 AC-Susceptibility Measurements

4.2.1 $\chi_{ac}(T)$-Temperature Sweeps

The susceptibility temperature sweeps $\chi(T)$ have been recorded at a constant field $H_{dc}$ by cycling the temperature from an initial value $T_i = 10$ K to an end value $T_f = 100$ K $> T_c$ (ZFC-curves) and back to $T_i$ (FC-curves) with a sweep rate of $dT/dt = 1$ K/10 min. As the hysteresis between the ZFC- and the FC-curves has been observed to be negligible and hidden within the noise of these measurements, in the following only the ZFC-curves are shown. In figure 4.1 typical $\chi'(T)$ and $\chi''(T)$ data for different constant applied magnetic fields $0 \leq H_{dc} \leq 800$ Oe are presented, while in figure 4.2, the $\chi'(T)$ and $\chi''(T)$ susceptibilities performed at $H_{dc} = 300$ Oe are further illustrated.

The susceptibility measurements $\chi(T)$ performed at different magnetic fields show in the low temperature regime a diamagnetic behaviour. Increasing temperature, a deviation from this behaviour is observed at almost the same temperature $T_{DL}(H)$ in all the $\chi(T)$ data. While for fields $H_{dc} \gtrsim 400$ Oe a smooth but evident increase in the inductive component $\chi'(T)$ an $\chi''(T)$ is detected (see figure 4.1), for fields $80$ Oe $\lesssim H_{dc} \lesssim 400$ Oe the increase in $\chi'(T)$ is more step-like and in $\chi''(T)$ a peak is measured (see figure 4.2). For fields $H_{dc} < 80$ Oe this feature is no more observed.

The increase of the inductive component $\chi'(T)$ at the temperature $T_{DL}(H)$ can be related within the "diffusive model" (Appendix A) to an increase of the flux flow resistivity $\rho_F(T)$. As an increase of resistivity can be associated to an increase of the vortex motion, which on his side can be related to a decrease of pinning, $T_{DL}(H)$ may be associated to a temperature above which pinning becomes weak. This in agreement with several measurements [3, 8, 9] showing at a temperature $T_d \simeq 40$ K and fields $H \gtrsim 500$ Oe a decrease of strong pinning upon increasing temperature. This so called depinning temperature $T_d$ has been observed to be field independent [3, 8, 9], in accordance with our results (see figure 4.3).

In all $\chi(T)$ measurements an evident crossover of the inductive component to $\chi' = 0$ accompanied by a sharp peak in the dissipative component $\chi''(T)$ is further observed (see figure 4.1 and 4.2). The temperature $T_{IL}(H)$ has been determined by the position of the peak in $\chi''(T)$ which correspond to the middle of the transition
Figure 4.1: $\chi'(T)$ and $\chi''(T)$ performed at different magnetic fields. The numbers indicate the applied field in Oe. The susceptibility $\chi'$ has been renormalized using the minimal value of the susceptibility measured for $H_{dc} = 0$ at the lowest temperature: $\chi'_0 = \chi'_{T \to 0}(H_{dc} = 0)$. 
Figure 4.2: $\chi'(T)$ and $\chi''(T)$ performed at $H_{dc} = 300$ Oe. The temperature $T_{DL}(T)$ corresponds to the middle of the small step-like transition in $\chi'$ at low temperatures, the temperature $T_{ML}$ coincides with the middle of a step like transition in $\chi'$ at intermediate temperatures, while the temperature $T_{IL}$ corresponds to the position of the peak in $\chi''$. 
in $\chi'(T)$. As shown in figure 4.3, this susceptibility transition is strongly field dependent. The downward shift of the transition temperature $T_{IL}(H)$ with increasing field is most pronounced at the lowest fields.

As discussed in literature [82,102,104], we identified the temperature $T_{IL}(H)$ of the peak in the dissipative component $\chi''(T)$ with the temperature where the irreversibility transition takes place. This irreversibility transition describes the crossover from an ‘irreversible state’, where the vortices have to overcome surface and geometrical barriers to enter and exit the sample, to a ‘reversible state’, where the vortices are free to move in and out of the specimen (section 2.3.3).
Furthermore, in the $\chi(T)$ measurements performed at magnetic fields $300 \text{ Oe} \lesssim H_{dc} \lesssim 350 \text{ Oe}$ a weak step-like increase in the inductive component $\chi'(T)$ and in the dissipative component $\chi''(T)$ at a temperature $T_{ML}(H)$ has been observed (see figure 4.2). As this step-like increase is more evident in the same field and temperature regime in the field sweep $\chi'(H)$ and $\chi''(H)$ data, an interpretation of this increase is given in the next section.

### 4.2.2 $\chi_{ac}(H)$-Field Sweeps

The susceptibility field sweeps $\chi(H)$ have been recorded at different constant temperatures by cycling the external field to a maximal value $H_{max} = 800 \text{ Oe}$ and back to zero with a typical sweep rate of $dH/dt = 3.1 \times 10^{-2} \text{ Oe/s}$. In this section only the $\chi(H)$ measurements on increasing the field are illustrated, as in this small field regime, hysteresis in the ac-signal was negligible and hidden within the noise of these measurements. In figure 4.4 and 4.5 typical $\chi'(H)$ and $\chi''(H)$ data measured at different temperatures in the regimes $25 \text{ K} \leq T \leq 60 \text{ K}$ and $40 \text{ K} \leq T \leq 85 \text{ K}$ are presented while the susceptibility measurements performed at $T = 32 \text{ K}$, $45 \text{ K}$ and $64 \text{ K}$ are further shown in figure 4.6, 4.7 and 4.8 respectively. The $H$-$T$ diagram depicted in figure 4.9 summarizes and compares the results obtained from susceptibility measurements $\chi(H)$ and $\chi(T)$.

Increasing the magnetic field, all susceptibility field sweep $\chi'(H)$ and $\chi''(H)$ data show at a certain low magnetic field $H_p(T)$, a sharp step-like increase from a initial constant value $\chi' / |\chi_0'| = -1$ and $\chi'' = 0$ respectively (see figure 4.4). When a small magnetic field ($H < H_p(T)$) is applied on a superconducting sample, the field is shielded by the Meissner currents and we expect $\chi' / |\chi_0'| = -1$ and $\chi'' = 0$. Accordingly, we interpreted $H_p(T)$ as the minimal field where vortices start to enter into the sample. In the absence of activation barriers for vortex penetration (section 2.3.3) this field of first flux penetration $H_p(T)$ correspond to the thermodynamic lower critical field $H_{cl}(T)$. However, for typical high temperature superconductor single crystals, surface and geometrical barriers or bulk pinning lead to values $H_p(T) > H_{cl}(T)$ [73].
4. Low Magnetic Field Diagram of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$+s

Figure 4.4: $\chi'(H)$ and $\chi''(H)$ performed at different temperatures $25 \leq T \leq 60$ K. The numbers indicate the temperature in K. The susceptibility $\chi'$ has been renormalized using the minimal value of the susceptibility measured at the temperature $T = 85$ K and zero magnetic field: $\chi'_o = \chi'_{T = 85 \text{ K}}(H = 0)$. 
4.2 AC-Susceptibility Measurements

Figure 4.5: $\chi'(H)$ and $\chi''(H)$ performed at different temperatures $40 \, K \leq T < T_c$. The numbers indicate the temperature in K. The susceptibility $\chi'$ has been renormalized using the minimal value of the susceptibility measured at the temperature $T = 85 \, K$ and zero magnetic field: $\chi'_0 = \chi'_T = 85 \, K(H = 0)$. 
Figure 4.6: $\chi'(H)$ and $\chi''(H)$ performed at the temperature $T = 32$ K. The field $H_p$ corresponds to the onset of the first increase of $\chi'/|\chi_0'|$ from the value -1 while the field $H_{13D}$ and $H_{SP}$ correspond to the local maximum respectively the local minimum in the $\chi'(H)$-curve.
4.2 AC-Susceptibility Measurements

Figure 4.7: $\chi'(H)$ and $\chi''(H)$ performed at the temperature $T = 45$ K. The field $H_p$ correspond to the onset of the first increase of $\chi'/|\chi'|$ from the value -1, the field $H_{13D}$ coincides with the position of an evident change of slope in the $\chi'(H)$-curve (see also inset) while the field $H_{ML}$ correspond to the middle of a step-like transition in $\chi'$. The dotted lines in the inset are guides to the eyes.
Figure 4.8: $\chi'(H)$ and $\chi''(H)$ performed at the temperature $T = 64$ K. The field $H_p$ correspond to the onset of the first increase of $\chi'/|\chi_o|$ from the value -1, the field $H_{13D}$ coincides with the position of an evident change of slope in the $\chi'(H)$-curve (see also inset), the field $H_{ML}$ correspond to the middle of a step-like transition in $\chi'$, while the field $H_{IL}$ correspond to the position of the peak in $\chi''$. The dotted lines are guides to the eyes.
After the step-like transition at $H_p(T)$, the $\chi'(H)$ curves in the temperature regime $25 \text{ K} \lesssim T \lesssim 36 \text{ K}$ show an evident change of slope at a field $H_{13D}(T)$ and a local minimum at a field $H_{SP}(T)$ (see figure 4.4 and 4.6). This decrease of the inductive component $\chi'$ in the field regime $H_{13D}(T) \lesssim H \lesssim H_{SP}(T)$ is in good agreement with a sudden increase of the bulk critical current density $j_c$ measured in the same temperature and field regime on Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ single crystals by global magnetization measurements [7], local magnetization measurements [6] and by permeability measurements [11]. This abrupt increase of the critical current density at low fields and temperatures is known as second peak transition (section 2.3.2). The same behaviour of the curves as observed in the inductive component $\chi'(H)$ are also measured in the dissipative component $\chi''(H)$ (see figure 4.4 and 4.6).

While the local minimum at $H_{SP}(T)$ in the $\chi'(H)$ measurements is no more detectable for temperatures $T \gtrsim 36 \text{ K}$, an evident change in slope at $H_{13D}(T)$ is further well observed (see figure 4.4). For temperatures $T \gtrsim 36 \text{ K}$ namely, an evident reduction of the increase of $\chi'(H)$ is detected at $H_{13D}(T)$ (see figure 4.7). However, increasing the temperature at which the $\chi'(H)$ curves are recorded, this change of slope becomes less evident (see figure 4.8), being no more visible for measurements performed at $T > 67 \text{ K}$. As discussed before, within the ‘diffusive model’ (Appendix A) a modification of the behaviour of the inductive component $\chi'(H)$ can be related to a variation in the pinning behaviour. Accordingly, the evident change in slope of the inductive component at the field $H_{13D}(T)$ may correspond to a crossover between different pinning regimes.

In addition to the change of slope at $H_{13D}(T)$, all the $\chi'(H)$ measurements performed in the temperature regime $38 \text{ K} \lesssim T \lesssim 64 \text{ K}$ show a well observable step-like behaviour at a field $H_{ML}(T)$ (see figure 4.5 and 4.7), where the field $H_{ML}(T)$ has been determined as the middle of the transition. Within the “diffusive model” (Appendix A) an increase of the inductive component $\chi'$ is related to an increase of the resistivity of the sample. As in the same field and temperature regime, transport measurements show a sharp increase in the resistivity which occur concurrently with the first-order vortex-lattice phase transition [105–107], it is a good assumption to associate the observed increase of $\chi'(H)$ at the field $H_{ML}(T)$ to this vortex melting transition (section 2.3.1). However, since this melting transition
Figure 4.9: Symbols denote: (*) field of first flux penetration \(H_p(T)\), (O) and (●) irreversibility transition field \(H_{IL}(T)\), (□) and (■) melting transition field \(H_{ML}(T)\), (●) second peak transition field \(H_{SP}(T)\), (∆) and (▲) depinning field \(H_{DL}(T)\) and (▼) proposed crossover field \(H_{13D}(T)\) between different pinning regimes. The open/closed symbols have been determinate by susceptibility temperature/field sweep measurements respectively. The fine dashed line are connections between the points. The demagnetization factor \(D \approx 0.68\) has been neglected.

is of first order \([5,34]\), one would expect a discontinuous step in \(\chi'(H)\) as observed at the transition field in local magnetization measurements as taken with miniature Hall-probes \([5]\). The wide transition in our measurements is related to the fact, that global measurements average over regions of different values of \(M\), and therefore different values of \(j(M)\) across the sample. As the external magnetic field is increased up to the melting transition (for simplicity we neglect the effects of geometrical barriers) a liquid front is formed at the sample border, proceeding towards the sample center when the field is further increased. For global measurement as in our case, the melting transition is therefore not observed at a characteristic field \(H\), but over a field range \(\delta H\) which depends on the vortex density gradient inside the
sample. An analogous explanation is also valid for temperature sweeps at a constant field discussed in the previous section. As in the inductive component $\chi'(H)$ curves, we observe a step-like behaviour at the melting transition also in the dissipative component $\chi''(H)$ (see figure 4.7).

For temperatures $50 \text{ K} \lesssim T < T_c$, the evident crossover of the inductive component to $\chi' = 0$ accompanied by a peak in the dissipative component $\chi''(T)$ previously associated to the irreversibility transition, is also observed in the field sweep susceptibility $\chi(H)$ data (see figure 4.5). As shown in figure 4.9, the values for the irreversibility transition field $H_{IL}(T)$ determined by $\chi(H)$ data are in good agreement with the values obtained by temperature sweep measurements $\chi(T)$. 
4.3 DC-Magnetization Measurements

To be able to compare the susceptibility with the global magnetization measurements, the magnetization $M(T)$ and $M(H)$ data have been recorded with the same $dT/dt$ and $dH/dt$ rates respectively, as the susceptibility $\chi(T)$ and the $\chi(H)$ data. In section 4.3.1 we will present the temperature sweep magnetization $M(T)$ measurements while in section 4.3.2 the field sweep $M(H)$ measurements are shown.

4.3.1 $M_{dc}(T)$-Temperature sweeps

In figure 4.10 typical ZFC-$M(T)$ data as a function of temperature for different constant applied magnetic fields $0 \leq H_{dc} \leq 800$ Oe are illustrated. As one can observe, all the ZFC-curves $M(T)$ show an evident increase of the magnetization at an almost field independent temperature $T_{0D} \simeq 22$ K, where $T_{0D}$ has been determined by the position of the maximal slope at the crossover (position of the peak in $dM/dT$). This remarkable behaviour of the magnetization $M(T)$ is in good agreement with several relaxation experiments showing a substantial decrease of the critical current density in the temperature regime up to $T \simeq 20$ K [10,108–110]. Moreover, as shown by Nideröst et al. [10], the collective pinning length $L_c = \xi(j_o/j_c)^{1/2} \simeq 4$ Å turns out to be for $T \lesssim 20$ K much smaller than the interlayer distance $d = 15$ Å, indicating that in the low temperature regime pinning involves elementary pancake vortices. This fact is reflected in our results, as for $T < 20$ K the magnetization curves show a practically diamagnetic behaviour (see figure 4.10) which is an indication of rather strong pinning as predicted for the single pancake vortex regime. Accordingly, it is reasonable to associate $T_{0D}$ as the crossover from the zero dimensional regime at low temperatures, to a higher dimensionality regime.

As further shown in the inset of figure 4.10, all the ZFC-$M(T)$ magnetization data reach $M = 0$ at a field independent temperature $T \simeq 93$ K $\simeq T_c$ (see figure 4.12). We associate this temperature with the transition from the superconducting to the normal state at the upper critical temperature $T_{Hc2}(H)$. 
Figure 4.10: Magnetization temperature sweeps $M(T)$ performed at different magnetic fields. The $M(T)$ data have been normalized by the corresponding constant applied magnetic field. The numbers indicate the applied field in Oe. The dashed line corresponds to the crossover to $M = 0$ at the temperature $T_{Hc2} \approx 93$ K.

In figure 4.11 the ZFC- and the FC-$M(T)$ data performed at $H_{dc} = 500$ Oe and $300$ Oe are further presented. The bold dashed lines correspond to the values of the depinning $T_{DL}(H)$, the melting $T_{ML}(H)$ and the irreversibility $T_{IL}(H)$ temperatures as obtained by susceptibility measurements $\chi(T)$ at the same applied fields. All the ZFC-$M(T)$ curves performed at $H_{dc} \gtrsim 400$ Oe show an evident reduced slope for $T \geq T_{DL}(H)$ (see inset figure 4.11 (a)). This reduction of the decrease of the current density $j(T) \propto |M(T)|$ around the depinning temperature is in good agreement with the variation of the pinning behaviour at $T_{DL}(H)$ as observed in the $\chi'(T)$ data (see section 4.2.1). However, this change of slope in the $M(T)$ curves measured at fields $H_{dc} \lesssim 400$ Oe becomes less observable as compared to the rather evident change of slope at the melting temperature $T_{ML}(H)$ (see inset...
Figure 4.11: Zero field cooling and field cooling magnetization measurements $M(T)$ (●) performed at $H_{dc} = 500$ Oe (a) and at $H_{dc} = 300$ Oe (b). The temperature $T_{OD}$ correspond to the peak in the derivative $d(M/H)/dT$ (○) while the temperature $T_{Hc2}$ coincides with the onset to $M = 0$. The temperatures $T_{DL}$, $T_{ML}$ and $T_{IL}$ (bold dashed lines) have been determined by the susceptibility measurements at the same applied fields (section 4.2.1).
4.3 DC-Magnetization Measurements

Figure 4.12: Symbols denote: (■) zero dimensional limit transition temperature $T_{0D}(H)$ and (○) temperature of the upper critical transition $T_{c2}(H)$ determined by magnetization measurements $M(T)$. The open symbols denote the crossovers/transitions determined by susceptibility measurements $\chi(T)$ and $\chi(H)$ as shown in figure 4.9. The demagnetization factor $D \approx 0.68$ has been neglected.

This is in accordance with the $\chi(T)$ measurements, where the crossover in $\chi'(T)$ at the temperature $T_{DL}(H)$ for magnetic fields $H_{dc} \lesssim 400$ Oe is very weak compared to the strong step-like transition at the melting temperature (see figure 4.2).

In magnetization $M(T)$ data, the irreversibility temperature $T_{IL}$ is typically determined by the onset of hysteresis. However, as the zero field cooling $M^{ZFC}(T)$ and the field cooling $M^{FC}(T)$ are smoothly joining curves, the exact position of the irreversibility temperature $T_{IL}(H)$ is not well determined by magnetization measurements. In any case, the value of $T_{IL}(H)$ as determined by our susceptibility data almost coincides with the onset of hysteresis as shown in the magnetization curve of figure 4.11.
4.3.2 $M_{dc}(H)$-Field sweeps

Typical magnetization curves $M(H)$ measured with a cycling rate $dH/dt \approx 3.10^{-2}$ Oe/s are given in figure 4.13. All the $M(H)$ measurements show a well defined Meissner regime at low fields, where the sample is in a completely diamagnetic state ($B = 0$ inside the specimen) and the initial slope of the curves is consequently $-4\pi M/H = 1$. At the field of first flux penetration $H_p$, the vortices enter into the sample and the magnetization curves deviate from the linear behaviour. As shown in figure 4.14, the values of the so determined field of first flux penetration $H_p(T)$ are in good agreement with the values determined by susceptibility measurements $\chi(H)$.

However, the values of $H_p(T)$ are strongly cycling rate $dH/dt$ dependent [2], as shown in figure 4.15 where $M(H)$-curves measured at the same temperature $T = 25$ K but at different cycling rates are given. As the investigated sample has a rectangular cross section, it was not possible to measure the lower critical field $H_{c1}(T)$ by decreasing the cycling rate $dH/dt$, as performed by Nideröst et al. [2] on a specimen with ellipsoidal-shaped cross sections. In this last case namely, geometrical barriers are not effective [4,44] and the lower critical field $H_{c1}(T)$ can be determined choosing a very slow cycling rate $dH/dT$, where vortices have been observed to creep over the relevant Bean-Livingston surface barriers [2].

The weak curvature of $H_p(T)$ for temperatures $T \lesssim 37$ K (see figure 4.12), has also been detected in other strongly anisotropic high temperature superconductors, such as Tl$_2$Ba$_2$CaCu$_2$O$_{8+\delta}$ [7], Pb$_2$Sr$_2$R$_{1-z}$Ca$_z$Cu$_3$O$_{8+\delta}$ [111] and Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ [2,3,7] in the configuration $H \perp ab$. Nideröst et al. [2] showed that this upturn can be explained in terms of measurements, where the system is out of equilibrium due to barriers for vortex penetration.

As illustrated in figure 4.13 (a) and 4.15, the magnetization curves in the temperature regime $20$ K $< T < 35$ K show an increase of $|M(H)|$ starting at about $H \approx 230$ Oe. Further, the magnetization changes at this field in opposite direction by increasing and decreasing fields. The behaviour of the magnetization curves in this field-temperature regime is known as second peak effect (section 2.3.2) and is related to an abrupt upturn in the bulk critical current density $j_c$ [6,7,11]. As shown in figure 4.15, the shape of the magnetization curves depends strongly
4.3 DC-Magnetization Measurements

Figure 4.13: Magnetization curves measured with a cycling rate \( \frac{dH}{dt} \sim 3.10^{-2} \) Oe/s. The numbers indicate the temperatures in K.
on the cycling rate $dH/dt$. However, the field regime where the second peak effect is observed in our magnetization curves $M(H)$ does not to depend on the cycling rate $dH/dT$. Moreover, the temperature regime $20 \, K < T < 35 \, K$ and the field regime $200 \, Oe \lesssim H \lesssim 400 \, Oe$ where the second peak effect in the magnetization curves has been detected, is in good agreement with the $H$-$T$ regime where an evident decrease of $\chi'(H)$ associated to an increase of $j$ has been observed in the susceptibility measurements.
Figure 4.15: Magnetization curves measured at $T = 25$ K and different cycling rates $dH/dt$. The arrows indicate the corresponding position of the field of first flux penetration $H_p$ where the magnetization deviates from the linear increase. To remark is that the second peak effect becomes less clear for fast cycling rates $dH/dt$. 

$\frac{dH}{dt} = 0.034$ Oe/s

$\frac{dH}{dt} = 0.026$ Oe/s

$\frac{dH}{dt} = 0.011$ Oe/s
4. Low Magnetic Field Diagram of Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$

The results obtained from susceptibility and magnetization measurements presented in the previous sections are summarized in the $H$-$T$ diagram depicted in figure 4.16. In the following we will discuss all the phase transitions and crossovers shown in figure 4.16 separately and we will try to relate them to the respective theoretical models discussed in section 2.3 and 2.4.

The field of first flux penetration $H_p(T)$ has been determined by susceptibility $\chi(H)$ (□) and magnetization $M(H)$ (○) field sweep measurements. In the absence of activation barriers for vortex penetration the field of first flux penetration corresponds to the thermodynamic lower critical field $H_{c1}$. For typical HTSC single crystals however, surface and geometrical barriers (section 2.3.3) or bulk pinning can lead to values of $H_p$ which lie somewhere in between the lower critical field $H_{c1} = (\Phi_0/4\pi\lambda_{ab}^2) \ln \kappa$ and the critical field $H_c = \Phi_0/2\sqrt{2}\pi\lambda\xi$. This means that the surface pinning barriers for vortex entry can preserve the Meissner state inside the specimen up to magnetic fields $H > H_{c1}$.

The upper critical field $H_{c2}(T)$ (○) has been determined by the crossover to $M = 0$ in the magnetization measurements $M(T)$. For $H \leq 800$ Oe, the temperature $T_{Hc2} \approx 93$ K at which this crossover takes place is field independent. This is in good agreement with the fact, that the investigated field regime is much below the upper critical field $H_{c2}(0) \sim 10^6$ Oe estimated by [1,91], so that $T_{Hc2} \approx T_c$ in the regime quoted above.

The irreversibility transition field $H_{IL}(T)$ (●) has been determined by the position of the peak in the dissipative component $\chi''$ in the susceptibility measurements $\chi(T)$ and $\chi(H)$. The irreversibility transition is related to Bean-Livingston surface barriers [42,43] and geometrical barriers [44] which inhibit the vortex entry (see section 2.3.3). Since geometrical barriers are expected to become active at high temperatures [3], at intermediate temperatures the vortex penetration is determined by Bean-Livingston surface barrier and is predicted to result in an exponential temperature dependence of the irreversibility line: $H_{IL}(T) \propto (T_0/2T) \exp(-2T/T_0)$ where $T_0 \approx \epsilon_0d/30$ and $\epsilon_0 = (\Phi_0/4\pi\lambda)^2$. This temperature dependence is in good agreement with the temperature behaviour of the measured irreversibility transition field,
and the fit of $H_{IL}(T)$ in the temperature regime $50 \, \text{K} \lesssim T \lesssim 72 \, \text{K}$ shown in figure 4.16, results in $T_0 = 23 \, \text{K}$. Assuming an interlayer distance for $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ of $d = 15 \, \text{Å}$ [81, 82], we obtain for the penetration depth $\lambda_{ab} \simeq 2000 \, \text{Å}$ which is also in agreement with values in literature (section 3.3).

The melting transition field $H_{ML}(T)$ (■) has been determined by a step-like crossover in the inductive component $\chi'$ and in the dissipative component $\chi''$ in the susceptibility measurements $\chi(T)$ and $\chi(H)$. As discussed in section 2.3.1, the Lindemann criterion provides a reasonable estimate for the melting field $H_{ML}(T)$ [26]. For HTSC the so estimated temperature behaviour of the melting transition is given by $H_m(T) = \beta_m (c_L^4/G_i) H_{c2}(0) (1 - T/T_c)^{\alpha}$ [26], where the power law exponent is $\alpha = 1$ for temperatures below $T_c$ and $\alpha = 2$ for temperatures near $T_c$ (section 2.3.1). Further, $c_L \simeq 0.15$ is the Lindemann number, $G_i$ is the Ginzburg number ($G_i \simeq 1$ for $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ [26]) and $\beta_m \simeq 5.6$. The curve in figure 4.16 is a best fit to the data resulting in $\alpha = 1.43$, which lie in between the value predicted for temperatures below and near $T_c$, and is in good agreement with power-law exponents measured by [5, 56, 57]. Further we obtain $H_{c2}(0) = 49 \, \text{T}$ (taking into account the demagnetization factor $D \simeq 0.68$) which is in the same order of magnitude with existing estimates [1, 91].

We want to remark here, that in spite of the favorable experimental conditions, namely a ultra sensitive experimental arrangement and a high quality $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ single crystal, we have not been able to observe any evidence indicating the presence of a narrow vortex-liquid phase just above the Meissner state as predicted theoretically (section 2.3.2).

The second peak transition field $H_{SP}(T)$ (♦) has been determined by the position of a local minimum in the inductive component $\chi'$ detected in susceptibility measurements $\chi(H)$ performed in a temperature regime $25 \, \text{K} \lesssim T \lesssim 36 \, \text{K}$. As shown in figure 4.16, the second peak transition field $H_{SP}(T)$ increases slightly with increasing temperature, joining the melting transition line $H_{ML}(T)$ at a so called critical temperature $T_{cr} \simeq 37 \, \text{K}$ and field $H_{cr} \simeq 400 \, \text{Oe}$. Several models have been proposed to explain the origin of this second peak transition, but there is still no general accepted theory which can describe this abrupt enhancement of the critical current density. However, the temperature dependence of $H_{SP}(T)$ is in agreement
with the upward curvature of the second peak transition predicted by Ertas and Nelson [41] for a disorder induced entanglement transition (section 2.3.2). Nevertheless, the equation 2.14 for this entanglement transition does not fit our results. Below $T \lesssim 22$ K we can no more observe the presence of a second peak transition (see figure 4.13), which is consistent with measurements reported in literature [6,11].

The almost field independent transition temperature $T_{0D}(H) \approx 22$ K (*) has been determined by the maximum slope of a large increase of magnetization in the temperature sweep measurements $M(T)$. As discussed in section 4.3.1, this strong decrease of $j(T)$ at low temperatures may correspond to a crossover from a strong pinned pancake vortex regime to a regime of weaker pinning in agreement with several relaxation experiments [10,108-110]. Within the pinning diagram for discrete layered superconductors proposed recently by Wagner et al. [12-14] and presented in section 2.4.3.1, this decrease of $j(T)$ measured at $T_{0D}(H)$ is in good agreement with a predicted low temperature crossover from a (0-0)D glassy regime, where pancake are strongly pinned individually, to a (0-1)D (for low fields) or a (0-3)D regime (for high fields), where thermal fluctuations become relevant and the critical current density $j_c(T)$ decreases exponentially with increasing temperature [12,13]. Moreover, the theoretically predicted zero dimensional limit temperature $T_g$, is not field dependent, which is in agreement with our measurements. In addition, assuming for the BSCCO compound the parameters $d = 15$ Å, $\lambda = 1800$ Å, $\xi = 20$ Å, and $U_p = (j_c/j_0)(\epsilon_0 d) \approx 25$ K where $j_c/j_0 \approx 0.03$ has been estimated from experimental results presented in [109], one obtains for the field independent crossover temperature $T_g(H) \approx 15$ K [13,14], which is not too far from $T_{0D} \approx 22$ K.

For magnetic fields $400$ Oe $\lesssim H \lesssim 800$ Oe, the temperature of the depinning transition $T_{DL}(H)$ (▲) has been determined by the onset of an evident increase of the inductive and the dissipative component in the $\chi(T)$ measurements. As discussed in section 4.2.1, the increase in $\chi'(T)$ with temperature can be related to a crossover from a strong to a weak pinning regime in agreement with several measurements [3,8,9]. In the pinning diagram for discrete layered superconductors by Wagner et al. [12-14], the temperature $T_{DL}(H)$ can be accordingly associated with the pancake depinning temperature $T_{pc}$. This temperature $T_{pc}$ indicates namely a crossover from a regime where thermal smoothing is inhibited by strong pinning ((0-1)D or (0-3)D) to a weak collective pinning regime of vortex segments (1D) or
of vortex bundles (3D). Further, the pancake depinning transition temperature $T_{pc}$ is predicted to be field independent, in good agreement with the almost field independent temperature $T_{DL}(H)$ (see figure 4.16). Moreover, assuming for the BSCCO compound the same parameters as above, we obtain for the depinning temperature $T_{pc} \simeq 50 \text{ K}$, which is again close to $T_{DL} \simeq 37 \text{ K}$.

Our experimental arrangement allowed us to detect a small but evident step-like crossover at $T_{DL} \simeq 37 \text{ K}$ also in the $\chi'(T)$ measurements performed at magnetic fields below $H_{\sigma} \simeq 400 \text{ Oe}$. Recently, an indication of such a crossover for magnetic fields $H \leq H_{\sigma}$ has been obtained with local Hall probes measurements of the current flow in Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ single crystals by Fuchs et al. [9]. Their temperature sweep experiments show for fields $200 \text{ G} \lesssim B \lesssim B_{\sigma} \simeq 750 \text{ G}$ an onset of strong pinning as the temperature drops below $T \simeq 40 \text{ K}$. Accordingly, our susceptibility measurements $\chi(T)$ confirm such a low field $H \leq H_{\sigma}$ crossover at the depinning temperature. For $B < 200 \text{ G}$ however, their depinning temperature becomes field dependent in their experiments [9], increasing with decreasing fields. This behaviour of the depinning temperature is not in agreement with our measurements as $T_{DL}(H)$ is observed to be almost field independent also for low magnetic fields. However, this field independence of $T_{DL}(H)$ is once more in good agreement with the predicted field independence of the pancake depinning temperature $T_{pc}$.

The field $H_{13D}(T)$ (▼) has been determined by an evident change in slope of the inductive component $\chi'(H)$ in the susceptibility field sweep measurements at temperatures $25 \text{ K} \lesssim T \lesssim 67 \text{ K}$. As shown in figure 4.16, $H_{13D}(T)$ is only slightly temperature dependent in the regime $25 \text{ K} \lesssim T < 37 \text{ K}$, while for temperatures $T \gtrsim 37 \text{ K}$ the field $H_{13D}(T)$ decreases almost linearly with slope $dH_{13D}/dT = -3.7 \text{ Oe/K}$. To our knowledge, this is the first experimental indication of a crossover in this low field $H-T$ regime.

As discussed in section 4.2.2, the behaviour of $\chi'(H)$ at $H_{13D}(T)$ can be related to a transition between different pinning regimes. According to the pinning diagram for layered superconductors [12-14] shown in figure 2.3, the field $H_{13D}(T)$ may then correspond to the predicted crossover field $B_{13}(T)$ between the single vortex regime [(0-1)D and 1D] and the vortex bundle regime [(0-3)D and 3D]. Using the parameters for Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ introduced above, $B_{13}(T_{dp}) \simeq B_{que}$ should be around 10 G at a temperature $T_{dp} \simeq 78 \text{ K}$ [13,14], which is close to our extrapolated value...
\[ B_{13D}(78 \text{ K}) \approx H_{13D}(78 \text{ K}) - H_p(78 \text{ K}) \approx 24 \text{ G} \] (taking into account the demagnetization factor \( D \approx 0.68 \)). This crossover field \( B_{13}(T) \) is predicted to be accompanied by a steep decrease of the critical current density upon increasing field. In our measurements however, the behaviour of \( \chi'(H) \) with increasing field can be associated for temperatures \( T \gtrsim 36 \text{ K} \) to a reduction of the decrease of the current density \( j(H) \) at \( H_{13D}(T) \) while in the temperature regime \( 25 \text{ K} \leq T < 36 \text{ K} \) even to an increase of \( j(H) \) at \( H_{13D}(T) \). A possible explanation of this behaviour is that the creep rate in the regime \([0, 1)D \) and \( 1D \) is predicted to be strong. Accordingly, in the \([0, 1)D \) and \( 1D \) regime the detected current density \( j(H) \) is considerably smaller than the critical value \( j_c(H) \) and consequently one cannot expect a steep decrease of the current density at \( H_{13D}(T) \). Moreover, in the 3D regime where creep is predicted to be weaker, the reduced decrease of the current density \( j(H) \) for \( H > H_{13D}(T) \) and temperatures \( T \gtrsim 37 \text{ K} \) is in good agreement with the theoretically predicted behaviour of the critical current density \( j_c(H) \).
Figure 4.16: H-T diagram of Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ (H$_{dc} \perp$ ab-planes), constructed using the susceptibility and the magnetization measurements presented in this chapter. Symbols denote: (□) and (◇) field of first flux penetration $H_p(T)$ determinate by susceptibility and magnetization measurements respectively; (○) upper critical field $H_{c2}(T)$; (●) irreversibility transition field $H_{IL}(T)$; (■) melting transition field $H_{ML}(T)$; (★) second peak transition field $H_{SP}(T)$; (▲) depinning field $H_{DL}(T)$; (⋆) zero dimensional limit transition field $H_{0D}(T)$; (▼) possible single-vortex to vortex-bundle transition field $H_{13D}(T)$. The line are fits to the curve (see text) while the fine dashed line are connections between the points. The demagnetization factor $D \simeq 0.68$ has been neglected.
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5 Low Magnetic Field Diagram of 2H-NbSe₂

5.1 Introduction

In the last decade theoretical and experimental investigations on high temperature superconductors have shown that on increasing the temperature or the magnetic field the vortex lattice undergoes a melting transition from a solid-like to a liquid-like state at magnetic fields well below the fluctuation-dominated crossover to the normal state defined as the upper critical field $H_{c2}(T)$. Moreover, theoretical results by Nelson [25] and more recently by Blatter and Geshkenbein [27] predict in anisotropic and layered superconductors the existence of a narrow vortex liquid phase just above the lower critical field $H_{c1}(T)$. However, HTCS are not ideal candidates for the detection of this low field melting line, since the width of the low field vortex liquid phase is expected to be of a few Oersted [26]. Better candidates are the weak pinning, short penetration length systems, such as the anisotropic low temperature superconductor 2H-NbSe₂, where the width of the low field vortex liquid phase is estimated to be of about twelve Oersted.

The anisotropic low temperature superconductor niobium diselenide 2H-NbSe₂ has attracted a great deal of attention over the last few years largely because many of its properties lie in between those of conventional superconductors and the high temperature cuprates. In the last few years several results of susceptibility measurements at low magnetic fields on 2H-NbSe₂ single crystals have been presented [16,28] showing a dip feature in the inductive $\chi'(T)$ data slightly before $T_c(H)$. This feature
is related to an anomalous peak of the critical current density $j_c$ known as 'peak effect' (PE), as measured by transport measurements [15] at higher fields. Moreover, Ghosh et al. [16] observed between 150 Oe and 50 Oe a weak reentrant behaviour of the peak effect temperature $T_{PE}(H)$. Relying on the assumption that the PE phenomenon can be associated with the melting of the vortex lattice [29,30], they interpreted the decrease of $T_{PE}(H)$ at low magnetic fields as the first experimental indication of a reentrant vortex liquid phase as predicted by Nelson [25]. However, below 50 Oe the PE was no more visible in their susceptibility measurements.

The goal of our measurements was to investigate the PE with emphasis at the very low fields $H < 50$ Oe in order to determine the reentrant behaviour of $T_{PE}(H)$ in the regime very close to $H_{cl}(T)$. For this purpose, susceptibility and magnetization temperature sweep as well as field sweep measurements have been performed on a high quality $2H$-NbSe$_2$ single crystal. Further, in order to gain a better understanding of the low field phase diagram deduced from these measurements, the time relaxation of the remanent magnetization $M_{rem}$ in the reduced temperature regime $0.5 \lesssim T/T_c < 1$ has been investigated. These relaxation measurements cover a time window of five decades $10^{-1} \text{s} < t < 10^4 \text{s}$, so that the current density $j$ can be studied from values very close to $j_c$ down to values considerably smaller than $j_c$.

In the first two sections the susceptibility measurements $\chi(T)$ and $\chi(H)$ and the magnetization measurements $M(T)$ and $M(H)$ are presented, while in section 5.4 the time relaxation measurements of the remanent magnetization $M_{rem}$ are described. In section 5.5 the low field $H-T/T_c$ diagram obtained from this measurements is discussed and interpreted.

### 5.2 AC-Susceptibility Measurements

All data are obtained using our standard SQUID magnetic susceptibility system involving the response to a small amplitude ac-field $H_{ac}$ superimposed on a background dc-bias-field $H_{dc}$, both maintained parallel to the c-axis of the anisotropic $2H$-NbSe$_2$ single crystal. The measurements presented in this section have been performed with an ac-field of frequency $\omega_{ac} = 16$ Hz and amplitude $H_{ac} = 30$ mOe.
In section 5.2.1 the temperature sweep $\chi(T)$ measurements are presented while in section 5.2.2 the field sweep $\chi(H)$ measurements are described.

5.2.1 $\chi_{ac}(T)$-Temperature Sweeps

The susceptibility temperature sweeps $\chi(T)$ have been recorded at a constant field $H_{dc}$ by cycling the temperature from an initial value $T_i = 4.2$ K to an end value $T_f = 10$ K $> T_c$ (ZFC-curves) and back to $T_i$ (FC-curves) with a very slow sweep rate of $dT/dt = 1$ K/60 min. In figure 5.1 typical $\chi'(T)$ and $\chi''(T)$ data for different constant applied magnetic fields $0 \leq H_{dc} \leq 880$ Oe are shown while in figure 5.2 the $\chi'(T)$ and $\chi''(T)$ susceptibilities performed at $H_{dc} = 500$ Oe are illustrated.

As depicted in figure 5.1, the $\chi'(T)$ ZFC-curves performed at fields $H_{dc} \geq 250$ Oe display a clear negative peak before the crossover to the normal state. This negative peak in $\chi'(T)$ has been explained by the peak effect phenomenon [17, 28, 99]. This phenomenon is common to a variety of irreversible type II superconductors (e.g. Nb [112], Nb$_3$Ge [113], Mo$_3$Si [114], 2H-NbSe$_2$ [115–117] and YBa$_2$Cu$_3$O$_7$ [102]) and refers to the appearance of a peak in the critical current density $j_c$ either as a function of applied field in isothermal measurements or as a function of temperature in isofield measurements, generally near $H_{c2}(T)$. The parameterized field values (i.e. $H_{PE}(T/T_c)$ normalized with respect to $H_{c2}$) at which peak effect has been observed to occur in these different superconductors vary considerably [17, 113, 114]. However, the $H_{PE}(T)$ values reported in 2H-NbSe$_2$ by different workers via different techniques appear to overlap [17, 115–120] and for high fields $H_{PE}(T)$ is observed to decrease linearly with increasing temperature. The slope $dH_{PE}/dT$ depends on the orientation of the applied field with respect to the crystal axis and for $H \parallel c$ is reported to be $dH_{PE}/dT \simeq -7.5$ kOe/K [117]. This is in good agreement with our result, as the peak effect temperature $T_{PE}(H)$ for fields $H \geq 250$ Oe increase linearly with decreasing field (see figure 5.3) with a rate $dH_{PE}/dT = -7.4$ kOe/K. Nevertheless, for magnetic fields $H \lesssim 250$ Oe the peak effect is no more visible in our $\chi'(T)$ data (see figure 5.1).
Figure 5.1: $\chi'(T)$ and $\chi''(T)$ performed at different magnetic fields. The numbers indicate the applied field in Oe. The arrows in the inset locate the value of the peak effect temperature $T_{PE}(H)$. The susceptibility $\chi'$ has been renormalized using the minimal value of the susceptibility measured for $H_{dc} = 0$ at the lowest temperature: $\chi'_0 = \chi'_{T \rightarrow 4.2K}(H_{dc} = 0)$. 
Figure 5.2: $\chi'(T)$ and $\chi''(T)$ performed at $H_{dc} = 500$ Oe. The temperature $T_{PE}(T)$ correspond to the minimum of the negative peak in $\chi'$, the temperature $T_{IL}$ with the position of the main peak and $T_{Hc2}$ with the position of the smaller peak in $\chi''$. 

$H = 500$ Oe
Figure 5.3: $H$-$T/T_c$ diagram of $2H$-NbSe$_2$ ($H_{dc} \perp ab$-planes), constructed using the susceptibility $\chi(T)$ measurements presented in this section. Symbols denote: (●) peak effect field $H_{PE}(T)$, (■) field of irreversibility transition $H_{IL}(T)$ and (▲) upper critical field $H_{c2}(T)$. The demagnetization factor $D \approx 0.63$ has been neglected.

An evident sharp crossover of the inductive component to $\chi' = 0$ is further observed in all susceptibility measurements. This crossover is accompanied by two peaks in the dissipative component $\chi''(T)$ (see inset in figure 5.2). As will be shown in section 5.3, it is a good assumption to associate the position of the main peak to the irreversibility transition and the smaller peak on the right, to the crossover at the upper critical field $H_{c2}(T)$. For fields $H < 50$ Oe, the two peaks can not be distinguished anymore. As illustrated in figure 5.3, the temperature $T_{Hc2}(H)$ of the position of the smaller peak, increases linearly with decreasing field, extrapolating to the critical temperature $T_c$. 
5.2 AC-Susceptibility Measurements

In all the $\chi'(T)$ measurements an evident hysteretic behaviour between the ZFC- and the FC-curves is further observed (see figure 5.2). This hysteretic behaviour becomes more pronounced by increasing the field $H_{dc}$ at which the measurements have been recorded. The FC-state has been detected to be slightly more diamagnetic, implying a larger current density $j$, than the ZFC state. Investigating the PE phenomenon in 2H-NbSe$_2$ single crystal samples containing varying amounts of quenched disorder, Banerjee et al. [28] related this hysteretic behaviour to pinning. They did not measure any history dependence in a clean sample while in a low purity specimen the hysteretic behaviour has been observed to be very pronounced. Further, Henderson et al. [15] showed by transport measurements on a low purity specimen, that in FC-measurements the disordered flux line lattice freezes into a strongly pinned metastable configuration where the current density $j$ is higher than in the stable ordered configuration obtained by ZFC-measurements. This 'supercooling' of the disordered flux line lattice by a field-cooling procedure, where it remains metastable in the absence of driving currents, has been recently observed in several other experiments [18,121,122].

5.2.2 $\chi_{ac}(H)$-Field Sweeps

Susceptibility field sweeps $\chi(H)$ have been recorded at different constant temperatures by cycling the external field to a maximal value $H_{max} = 880$ Oe and back to zero with a typical cycling rate of $dH/dt = 3.10^{-2}$ Oe/s. In this section only the $\chi(H)$ measurements on increasing field are shown, as the hysteresis between the ZFC- and the FC-curves has been observed to be negligible and hidden within the noise of these measurements.

In figure 5.4 the $\chi'(H)$ and $\chi''(H)$ data recorded at different temperatures are illustrated. The evident negative peak as observed slightly before the crossover to the normal state in the $\chi'(T)$ measurements is not detected in the $\chi'(H)$ data. However, the inductive component recorded at $0.96 < T/T_c < 0.98$ show in the expected peak effect regime a reduced increase of $\chi'$ (see inset in figure 5.5). In the $H$-$T/T_c$ phase diagram illustrated in figure 5.6, the start- and end-field of this reduced increase are compared with the results of the temperature sweep measurements.
Figure 5.4: $\chi'(T)$ and $\chi''(T)$ performed at different temperatures. The numbers indicate the reduced temperature $T/T_c$. The susceptibility $\chi'$ has been renormalized using the minimal value of the susceptibility measured at $T/T_c = 0.994$ and zero magnetic field: $\chi'_0 = \chi'_{T/T_c=0.994}(H = 0)$. 
Figure 5.5: $\chi'(H)$ and $\chi''(H)$ performed at $T/T_c = 0.973$ Oe. The dashed line in the inset correspond to the starting respectively end-point of the regime where a reduced increase with field is observed. The temperature $T_{IL}$ coincides with the position of the main peak and $T_{Hc2}$ with the position of the smaller peak in $\chi''$. 
Figure 5.6: H-T/Tc diagram of 2H-NbSe2 (H_{dc} \perp ab-planes), comparing the result of the susceptibility \(\chi(T)\) with the results of the \(\chi(H)\) measurements. Symbols denote: (O) start and end-field of the plateau measured in the inductive \(\chi'(H)\) component, (●) peak effect field \(H_{PE}(T)\) measured by \(\chi'(T)\) measurements, (□) and (■) irreversibility line \(H_{IL}(T)\) and (△) and (▲) upper critical field \(H_{c2}(T)\). The closed/open symbols have been determined by susceptibility temperature/field sweep measurements respectively. The demagnetization factor \(D \simeq 0.63\) has been neglected.

The main and the secondary peak observed in the dissipative component of the temperature sweep measurements, are also detected in the field sweep measurements (see figure 5.4). As shown in figure 5.6, the position of the two peaks in \(\chi''(H)\) corresponding to the irreversibility transition field \(H_{IL}\) and the upper critical field \(H_{c2}\) are in good agreement with the result obtained by temperature sweep measurements.
5.3 DC-Magnetization Measurements

Magnetization measurements $M(T)$ and $M(H)$ have been recorded with the same $dT/dt$- and $dH/dt$-rates respectively, as the susceptibility measurements $\chi(T)$ and the $\chi(H)$. In section 5.3.1 we will present the temperature sweep magnetization $M(T)$ measurements while in section 5.3.2 the field sweep $M(H)$ measurements are described.

5.3.1 $M_{dc}(T)$-Temperature Sweeps

In figure 5.7 typical ZFC-$M(T)$ data normalized by the corresponding magnetic field $H_{dc}$ are plotted together while in figure 5.8 the normalized ZFC- and the FC-$M(T)$ curves performed at $H = 500$ Oe are illustrated.

As depicted in figure 5.7, the ZFC-$M(T)$ curves measured at magnetic fields $H > 42$ Oe show for $T/T_c \lesssim 0.92$ a linear increase with temperature. However, in the neighbourhood of $T_{Hc2}$ a sudden strong reduction of this increase in magnetization is observed (see figure 5.8 (b)). Moreover, this deviation from the linear behaviour at a temperature $T_{DT}$ is directly followed by a sharp increase of $M(T)$ at a temperature $T_{WP}$ (position of the main peak in the derivative $dM/dT$).

This behaviour of the ZFC-$M(T)$ curves in the neighbourhood of $T_{Hc2}$ can be well explained by a sudden increase of the critical current density $j_c$ as expected in the peak effect regime. The deviation from the linear increase of the magnetization at $T_{DT}$ can be associated to a change in slope in $j_c(T)$ as expected slightly below the peak effect temperature. The following reduced increase of the magnetization $M(T)$ can be explained by a sudden increase of $j_c(T)$ in the peak effect regime which consequently inhibits vortex entry. While the strong decrease of the magnetization $M(T)$ as measured for temperatures $T > T_{PE}^{de}$, where $T_{PE}^{de}$ is the onset of the strong decrease, can be associated to a decrease of $j_c$ after the peak effect temperature. The temperature $T_{WP}$ correspond to the maximal slope of this decrease. Actually, the temperature regime of the peak effect as determined by susceptibility measurements, coincides with the temperature regime where the deviation of the linear increase of $M(T)$ is observed. Moreover, the peak effect temperature $T_{PE}$ is in good agreement...
Figure 5.7: Figure (a) shows typical zero field cooled $M(T)$ data as a function of reduced temperature $T/T_c$. The $M(T)$ data have been normalized by the corresponding constant applied magnetic field $H_{dc}$. The numbers indicate the $H_{dc}$ field in Oe. Figure (b) is an expansion of the region indicated by the pointed rectangle in figure (a).
5.3 DC-Magnetization Measurements

Figure 5.8: Figure (a) show the ZFC- and FC-$M(T)$ data as a function of temperature, normalized by the constant applied magnetic field $H_{dc} = 499.7$ Oe. Figure (b) is an expansion of the region indicated by the pointed rectangle in figure (a). The dashed lines in figure (b) correspond to the temperatures determined as follows: $T_{DT}$ → the deviation from the linear increase of $M(T)$, $T_{PE}$ → onset of the strong increase of $M(T)$, $T_{WPT}$ → position of the maximal slope of the strong increase of $M(T)$, $T_{IL}$ → onset of hysteresis (see inset in figure (b)), $T_{Hc2}$ → value where the magnetization reaches the base line (see inset in figure (b)). The bold pointed line in figure (b) correspond to the peak effect.
with the temperature $T^\text{dc}_{\text{PE}}$ of the onset of the strong increase of $M(T)$ (bold pointed line in figure 5.8).

As further shown in figure 5.8 (b), slightly above the sharp increase of the magnetization at $T_{WPT}$, an onset of reversible behaviour is observed. This onset has been associated to the temperature $T_{IL}(H)$ of the irreversibility transition which also coincides with the position of the smaller peak in $dM/dT$ to the right of the main peak. By further increasing temperature, the magnetization reaches the base line at the temperature $T_{Hc2}(H)$.

The ZFC-measurements recorded at magnetic fields $H \leq 42$ Oe show a non linear increase of $M(T)$ in the low temperature regime (see figure 5.7). Assuming in this regime a linear decrease of the critical current density $j_c$ as measured by transport measurements at higher fields [15], this nonlinearity can be explained within the Bean model [123,124] by an undercritical state of the sample ($H < H^*$) at the starting temperature $T_i$. The onset of linearity in $M(T)$ is then observed as the applied field $H_{dc}$ fully penetrates the sample and the critical state is reached ($H \geq H^*$). For magnetic fields $H \geq 10$ Oe this linear behaviour of $M(T)$ is observed before the temperature reaches the investigated peak effect regime and the determination of the temperature $T_{DT}$ of the deviation from the linear increase is still reasonable (see figure 5.9 (b)). Further, also the sharp increase of the magnetization at $T_{WPT} \lesssim T_{Hc2}$ is still evident. However, for magnetic fields $H < 10$ Oe the above described behaviour of $M(T)$ in the neighbourhood of $T_{Hc2}$ is no more visible and only a crossover to the normal state is detected.

The results obtained by the magnetization measurements $M(T)$ recorded at different magnetic fields are summarized and compared with susceptibility measurements in figure 5.10. For $H \gtrsim 250$ Oe, the temperature $T_{DT}(H)$ of the sudden increase of $j_c$ and the temperature $T^\text{dc}_{\text{PE}}(H)$ of the onset of the subsequent strong decrease of $j_c$, increase linearly with decreasing field. As one can observe in figure 5.10, $T^\text{dc}_{\text{PE}}(H)$ is in good agreement with the peak effect temperature determined by susceptibility $\chi(T)$ measurements. For fields $85$ Oe $\lesssim H \lesssim 250$ Oe a reentrant behaviour of $T_{DT}(H)$ and $T^\text{dc}_{\text{PE}}(H)$ is observed, while for $H \lesssim 85$ Oe this two temperatures increase again with decreasing magnetic field. On the contrary, the temperature $T_{WPT}(H)$ of the maximal slope of the strong decrease of $j_c$ does not show any reentrant behaviour, increasing linearly with decreasing field and extrapolating.
Figure 5.9: Figure (a) shows the ZFC- and FC-M(T) data as a function of temperature, normalized by the constant applied magnetic field $H_{dc} = 30.4$ Oe. Figure (b) is an expansion of the region indicated by the pointed rectangle in figure (a). The dashed lines in figure (b) correspond to the same temperatures as described in figure 5.8.
to a temperature $T \simeq 0.988 T_c$. Moreover, the temperature of the irreversibility line $T_{IL}(H)$ lies slightly below the upper critical temperature $T_{Hc2}(H)$ and both increase monotonically with decreasing field. The temperatures $T_{IL}(H)$ and $T_{Hc2}(H)$ are in good agreement with the temperatures determined by the respective peaks in the dissipative component $\chi''$ of the susceptibility measurements. Hence, it is a good assumption to associate the main peak in $\chi''$ to the irreversibility transition and the smaller peak to the crossover to the normal state.
Figure 5.11: Figure (a) shows the ZFC- and FC-M(T) data as a function of temperature recorded on a second sample. The data has been normalized by the constant applied magnetic field $H_{dc} = 200.4$ Oe. In figure (b) the results of both samples are compared. The dashed lines are fitting curves to the point measured on the first sample.

The same results as presented above have been reproduced on a second $2H$-$\text{NbSe}_2$ sample of smaller size. In figure 5.11 (a) the temperature sweep magnetization data $M(T)$ recorded at a magnetic field $H = 200$ Oe are shown, while in figure 5.11 (b) results of both samples are summarized and compared.
5. Low Magnetic Field Diagram of 2H-NbSe$_2$

5.3.2 $M_{dc}(H)$-Field Sweeps

Figure 5.12 illustrates typical isothermal magnetization curves measured at different temperatures slightly below $T_c$. For low magnetic fields, these magnetization curves display a well defined Meissner regime. In this regime the sample is in a completely diamagnetic state ($B = 0$ inside the specimen) and the initial slope of the curves is consequently $-4\pi M/H = 1$ (see inset (1) in figure 5.12). At the field of first flux penetration $H_p \geq H_{c1}$, the vortices enter into the sample and the magnetization $M(H)$ deviate from the linear increase. As shown in the inset of figure 5.13, the so determined field of first flux penetration $H_p(T)$ show a linear temperature dependence in this temperature regime.

![Figure 5.12: Typical isothermal magnetization curves measured at different temperatures slightly below $T_c$. The numbers indicate the reduced temperature $T/T_c$ at which the curves have been recorded. Inset (1) is an expansion of the region indicated by the pointed rectangle while inset (2) show the difference magnetization ($= 4\pi (M(H \uparrow) - M(H \downarrow))$) values reflecting the field variation of the critical current density $j_c(H)$.](image-url)
Figure 5.13: In this figure the start (○) and the end-point (●) of the step-like decrease of $j_c(H)$ (see inset (2) in figure 5.12) are compared with the results obtained by the magnetization temperature sweeps $M(T)$ presented in the previous section. The inset show the linear temperature dependence of the field of first flux penetration $H_p(T)$ determined by the position where the magnetization $M(H)$ deviate from the linear increase (arrows in inset (1) of figure 5.12)

The inset (2) of figure 5.12 illustrates the field dependence of $4\pi \Delta M(H)$ (i.e. the difference in magnetization values at a given $H$ during the forward and reverse hysteresis runs) of the magnetization curves shown in figure 5.12. This magnetization hysteresis width is a measure of macroscopic currents set up within the sample and the field variation of $\Delta M(H)$ is proportional to the field variation of the critical current density $j_c(H)$ [123,124]. Accordingly, the magnetization curves measured in the reduced temperature regime $0.96 \lesssim T/T_c \lesssim 0.986$ show an evident step-like behaviour of $j_c(H)$. As illustrated in figure 5.13, the starting and the end-point of this decrease of $j_c(H)$ lies slightly below respectively above the $H_{WPT}(T)$ line determined by the strongest decrease of the critical current density as measured in the $M(T)$ curves. This is a further indication that at this line a sharp decrease of $j_c$ occurs. However, no increase in the magnetization curves has been observed at the peak effect field as estimated from susceptibility and magnetization measurements.
5.4 Time Relaxation of the non-Equilibrium Vortex State

In the following section, we investigate experimentally the low field vortex dynamics in $2H$-NbSe$_2$ single crystal for magnetic fields perpendicular to the $ab$-planes. The measurements of the relaxation of the remanent magnetization $M_{\text{rem}}$ are taken in the reduced temperature interval $0.52 \leq T/T_c \leq 0.99$ and cover a time window of six decades $10^{-1} \leq t \leq 10^5$ s.

In figure 5.14 and 5.16 the time dependence of the remanent magnetization $M_{\text{rem}}$ for a typical set of data are shown. Further, in figure 5.17 (a) and (b) the current density $j(t_s)$ at the starting time $t_s \approx 10^{-1}$ s and the fraction of the current density $j(t)/j(t_s)$ for different times respectively, are plotted as a function of reduced temperatures. The critical current density $j(t)$ has been obtained from the remanent magnetization $M_{\text{rem}}(t)$ by formula B.3 (see appendix B). As illustrated in figure 5.14 and 5.16, all relaxation measurements show a non-logarithmic behaviour. However, for reduced temperatures $T/T_c \lesssim 0.83$ the fraction of the remanent magnetization decreases with increasing temperature while for $T/T_c \gtrsim 0.83$ this fraction is observed to increase with temperature (see also figure 5.17 (b)). Further, as shown in figure 5.17 (a), in the regime $T/T_c \lesssim 0.83$ the current density $j(t_s)$ measured at the starting time $t_s \approx 10^{-1}$ s decrease very slowly with increasing temperature while in the regime $T/T_c \gtrsim 0.83$ an almost strong decrease is detected. We will discuss these temperature regimes separately and distinguish them as follows: an intermediate temperature regime for $T/T_c \lesssim 0.83$ and a high temperature regime for $T/T_c \gtrsim 0.83$.

5.4.1 Intermediate Temperature Regime ($0.52 \lesssim T/T_c \lesssim 0.83$)

As shown by Maley et al. [125] it is possible to determine the activation barrier $U(j)$ for vortex motion directly from the relaxation data $j(t)$. The activation barrier $U(j)$ can be expressed by [125]

\[ U(j) \simeq -k_BT \ln \left( s \frac{\partial j}{\partial t} \right) + k_BT \ln \left( s \frac{j_c}{\tau_0} \right), \tag{5.1} \]
where \( s = 1 \text{ cm}^2/\text{s}/\text{A} \), \( j_c \) is the critical current density and \( \tau_0 \) is the inverse attempt frequency. Notice that the term \( k_B T \ln |s j_c/\tau_0| \) is independent of \( j \). Plotting the expression \( -k_B T \ln |s \partial j/\partial t| \) as a function of current density at different temperatures \( T \), a set of curves is found which are vertically shifted with respect to each other. For a temperature interval where the functional dependence between the activation barrier \( U \) and the current density \( j \) is essentially temperature independent, this shift is given by the term \( a \Delta T \), where \( a \approx \ln |s j_c/\tau_0| \) is a constant, and \( \Delta T = T_2 - T_1 \) is the temperature difference between two considered curves. Combining the data measured at different temperatures \( T \), the activation barrier \( U(j) \) is obtained over a wide current density range.

For the relaxation measurements performed at reduced temperatures \( T/T_c \lesssim 0.83 \) (see figure 5.14), the data which are obtained from the expression \( -k_B T \ln |s \partial j/\partial t| \) at different temperatures \( T \) have been accurately mapped onto a common curve using a single constant \( a \). Nevertheless, we have observed that the values obtained by \( -k_B T \ln |s \partial j/\partial t| \) were slightly tilted with respect to each other. Moreover, the constant \( a \approx \ln |s j_c/\tau_0| \approx 128 \) used for matching the curves lead to a value \( j_c/\tau_0 \) of the order of \( 10^{55} \text{ A/cm}^2 \text{s} \) which is too high for having any physical meaning. This may be related to the assumption, that the above described activation barrier \( U(j) \) does not depend explicitly on temperature. As a matter of fact, a better result is obtained introducing a temperature dependence of the activation barrier \( U \) \[126\],

\[
U(j) \left( \frac{1}{\beta} \left( 1 - \left( \frac{T}{T_c} \right)^\beta \right) \right)^{\alpha} \approx -k_B T \ln |s \partial j/\partial t| + k_B T \ln |s j_c/\tau_0|.
\]

Choosing \( \beta = 1 \) we noticed that the curves overlapped for an exponent \( \alpha = 1 \), while for \( \beta = 2 \) the curves matched for an exponent \( \alpha = 1.3 \). In addition, we found in both cases \( a \approx 42 \). By inserting this value into the expression \( a \approx \ln |s j_c/\tau_0| \) one obtains a judicious value for \( j_c/\tau_0 \) of the order of \( 10^{18} \text{ A/cm}^2 \text{s} \).
Figure 5.14: Normalized remanent magnetization vs. time, measured at reduced temperatures $T/T_c < 0.83$ after cycling the sample in an external magnetic field to a maximal value $H_{\text{max}} = 450$ Oe. The time origin is given by the instant, when the externally applied magnetic field $H$ starts being decreased and the $t_s \approx 10^{-1}$ s is the time, when the first point of the relaxation of $M_{\text{rem}}$ is taken. The numbers indicate the reduced temperatures $T/T_c$ at which the relaxation measurements have been performed.

In figure 5.15 the obtained potential $U(j)$ are shown. From the double logarithmic plot, one can observe that both activation barriers $U(j)$ follow a power-law behaviour. Fitting this potential with the general formula for the activation barrier $U(j) = U_c((j_c/j)^\beta - 1)$ [26], we found the values $U_c \approx 80 - 140$ K, $j_c(T = 0) \approx (8 - 9).10^3$ A/cm$^2$ and $\mu \approx 6$ for the temperature dependent potential with the exponent $\beta = 1$, $\alpha = 1$ and $\beta = 2$, $\alpha = 1.3$ respectively. The values of the critical current $j_c(T = 0)$ are of the same order of magnitude as the values found in literature [15,20,100]. However, the exponent $\mu \approx 6$ obtained for both potential is too high as compared to theory [26].
Figure 5.15: Figure (a): Flux creep activation barrier $U(j)$ for reduced temperatures $T/T_c \leq 0.83$ as determined from the magnetic relaxation data by the method of Maley et al. [125] with a temperature dependent potential $U(j,T) = U(j)((1/\beta)(1 - (T/T_c)\beta)^\alpha$ with $\beta = 2$, $\alpha = 1.3 \rightarrow$ curve (1) and $\beta = 1$, $\alpha = 1 \rightarrow$ curve (2) (the constant used for matching the curves is $\alpha = 42 \pm 1$). The horizontal segments represent the current windows as obtained from data at a fixed temperature. The lines are fit with a potential...
5.4.2 High Temperature Regime \((T/T_c \gtrsim 0.83)\)

In order to find the activation barrier \(U(j)\) for reduced temperatures \(T/T_c \gtrsim 0.83\), the relaxation data have been again evaluated with the method of Maley et al. Nevertheless, it was not possible to obtain a unique smooth curve by simply shifting the data obtained at different temperatures along the vertical axis as the curves were strongly tilted with respect to each other. Thus, within the temperature range \(T/T_c \gtrsim 0.83\), it was not possible to find a unique functional relation between \(U\) and \(j\) following the above approach.

The extremely non-logarithmic behaviour of the curves is a characteristic of relaxation measurements performed in the temperature regime \(T/T_c \gtrsim 0.83\) (see figure 5.16). Further, the relaxation measurements of the remanent magnetization performed at reduced temperatures \(T/T_c \gtrsim 0.92\) show, after an extremely fast decay in the first few seconds, a practically flat behaviour. This behaviour becomes more evident for temperatures close to \(T_c\). A possible interpretation of the vortex dynamics observed for this reduced temperatures \(T/T_c \gtrsim 0.92\) is the simultaneous occurrence into the sample of two different pinning regimes with different relaxation times. The high relaxation rates which are measured in the first few seconds are mainly the result of the strong decay in the regime where the vortices are less pinned. For longer times, most of the flux has left the sample and only the flux gradient density of the strongly pinned vortices remain. A more detailed discussion of the possible nature of this two different pinning regimes will be given in section 5.5.

In figure 5.17 (a) the current density \(j(t_s)\) for the starting time \(t_s \approx 10^{-1}\) s is plotted as a function of reduced temperature. As one can observe, in the regime \(T/T_c \gtrsim 0.83\) the current density \(j\) decreases rapidly with increasing temperature extrapolating to a temperature \(T = 0.988 T_c\) slightly below the critical temperature \(T_c\).
Figure 5.16: Normalized remanent magnetization vs. time, after cycling the sample in an external magnetic field. In parenthesis the values of the maximal cycling fields $H_{\text{max}}$ are given: $T/T_c \leq 0.93$ ($H_{\text{max}} \approx 450$ Oe) and $T/T_c \geq 0.93$ ($H_{\text{max}} \approx 100$ Oe). The numbers indicate the reduced temperatures $T/T_c$ at which the relaxation measurements have been performed. The time origin is given by the instant, when the externally applied magnetic field $H$ starts being decreased and the $t_s \approx 10^{-1}$ s is the time, when the first point of the relaxation of $M_{\text{rem}}$ is taken.
Figure 5.17: Figure (a): Current density $j(t_s)$ measured at the starting time $t_s = 10^{-1}$ s as a function of reduced temperature. The lines serve as guides to the eyes. Figure (b): Fraction of the current density $j(t)/j(t_s)$ as a function of reduced temperature, where $t_s = 10^{-1}$ s is the starting time. The numbers in the inset indicate the time at which the different current densities $j(t)$ have been determined.
5.5 Low Field $H$-$T$ Diagram of $2H$-NbSe$_2$

The results obtained in the previous section for the continuous layered $2H$-NbSe$_2$ single crystal in magnetic fields $H \perp ab$-planes are now summarized and discussed. In figure 5.18 the low field phase diagram deduced from our susceptibility and magnetization measurements with a qualitative interpretation of the different vortex states is shown.

For magnetic fields $H \gtrsim 250$ Oe, the peak effect temperature $T_{PE}(H)$ determined by the position of the negative peak in our susceptibility $\chi'(T)$ data, increases linearly with decreasing field in agreement with results in literature [16, 17, 117]. However, for $H < 250$ Oe the peak effect is no more visible in our susceptibility measurements. This is possibly related to the very low amplitude of the $ac$-field $H_{ac} = 30$ mOe, indicating that the peak effect in this low field regime might be triggered above certain amplitude of the detecting field [127].

In the magnetization measurements $M(T)$ the peak effect regime can also be detected for low magnetic fields $H \gtrsim 10$ Oe. As a matter of fact, an evident reduction of the linear increase of the magnetization directly followed by a strong decrease to $M \gtrsim 0$ is observed in the ZFC-$M(T)$ curves slightly below $T_{Hc2}(H)$. This behaviour can be well explained by a sudden peak in the critical current density as expected in the PE regime. Further, the peak effect temperature $T_{PE}^{dc}(H)$ as estimated from the magnetization measurements for fields $H \gtrsim 250$ Oe is in good agreement with the peak effect temperature $T_{PE}(H)$ as determined from our susceptibility data. Moreover, the temperature $T_{PE}^{dc}(H)$ as well as the temperature $T_{DT}(H)$ of the onset of the reduction in magnetization associated to the onset of the peak effect regime, show a reentrant behaviour for $85$ Oe $\lesssim H \lesssim 250$ Oe confirming the results of Ghosh et al. [16].

One mechanism to explain the sudden increase of $j_c$ at the peak effect [128] slightly below the upper critical field is the softening of the shear modulus of the vortex lattice as $H_{c2}(T)$ is approached. Lattice (shear) distortions which maximize the energy gain due to the pinning thus cost less elastic energy. As a consequence, $j_c$ increases since the flux line lattice can adapt better to its pinning environment. The shear modulus of the vortex lattice becomes also exponentially small at low field...
5. Low Magnetic Field Diagram of 2H-NbSe$_2$

values $H \gtrsim H_{cl}$ [26] where the vortex interactions start to decrease. Consequently, this leads to a reentrance of the peak effect regime for low magnetic fields, in agreement with our results for $85$ Oe $\lesssim H \lesssim 250$ Oe.

In the last few years several authors [29, 30, 116, 120] also proposed a rapid decrease of the shear modulus slightly below a flux line lattice melting transition. Accordingly, they suggested that the peak effect track the melting transition extremely closely. Relying on this assumption, Ghosh et al. [16] interpreted the weak reentrant behaviour of the peak effect temperature detected at low magnetic fields, as first experimental indication of a reentrant vortex liquid phase as theoretically predicted by Nelson [25]. An indication of a thermal melting transition is also observed in our magnetization $M(T)$ measurements by a strong decrease of the critical current density $j_c$ slightly above the estimated peak effect temperature $T^{dep}_P(H)$. However, the temperature $T_{WFT}(H)$ corresponding to the maximal slope of the decrease in $j_c(T)$, increases monotonically with decreasing field, showing no reentrant behaviour. Consequently, we do not interpret the reentrance of the peak effect as the reentrant vortex liquid phase as proposed by Ghosh et al. [16].

A further mechanism to explain the peak effect phenomenon has been recently proposed by Paltiel et al. [18]. They suggest a disorder-driven first order transition from an ordered Bragg Glass, which is dominated by the elastic energy, to a highly disordered solid which is governed by the pinning energy. The reentrance of the peak effect transition is then related to a rapid decrease of the elastic energy at low fields ($B \approx \Phi_0/\lambda^2$) where the vortex interactions start to decrease. Further, the commonly observed smearing of the peak effect in 2H-NbSe$_2$ crystals is explained by the injection of disordered vortices through the sample edges in the conventional strip configuration [129]. These features are found to be absent in transport studies in a Corbino disk geometry in which the circulating vortices do not cross the sample edges and a very sharp increase of $j_c$ at the peak effect is observed [18]. A number of anomalous instability phenomena reported in 2H-NbSe$_2$ in the last few years [15, 99, 122, 129–131] can be well explained by the mechanism suggested by Paltiel et al. [18].

According to this scenario, our measurements can be interpreted as follows (see figure 5.18): the increase of $j_c(T)$ observed at $T_{DT}(H)$ can be associated to a crossover from an ordered Bragg Glass to a metastable disordered state, formed dynamically
by an edge-contamination mechanism [129]. This is also consistent with neutron scattering measurements [122] where a structural evidence of a disordering of a well ordered lattice at the onset of the peak effect regime has been observed. While at the estimated peak effect temperature \( T_{PE}^{\text{pc}}(H) \), a phase transition to a stable highly disordered solid takes place. This is also in agreement with several experiments which observe at the peak effect temperature/field a crossover from a metastable to a stable state [15,129,131]. Nevertheless, the mechanism suggested by Paltiel et al. [18] does not explain the strong decrease of the critical current density into a weakly pinned state as observed in our magnetization \( M(T) \) measurements directly after the peak effect regime. Moreover, we have no appropriate explanation for the second increase of \( T_{DT}(H) \) and \( T_{PE}^{\text{pc}}(H) \) with decreasing field observed in the magnetic field regime \( H \lesssim 85 \text{ Oe} \).

A further indication of this second increase of the onset temperature \( T_{DT}(H) \) of the peak effect regime for low magnetic fields \( H \lesssim 85 \text{ Oe} \) is also given by our time relaxation measurements of the remanent magnetization \( M_{\text{rem}} \). The decay of \( M_{\text{rem}} \) performed at reduced temperatures \( T/T_{c} \gtrsim 0.92 \) shows namely, after an extremely fast decay in the first few seconds, a practically flat behaviour. A possible interpretation of the vortex dynamics in this regime is the simultaneous occurrence into the sample of two different pinning regimes with different critical current densities. This is in good agreement with the low field crossover from a pinning regime of low (e.g. Bragg Glass) to a pinning regime of higher \( j_{c} \) (e.g. highly disordered solid). The initial field profile in the sample may then consist of ‘disordered solid’ pinning in the central region of the sample and ‘Bragg Glass’ pinning close to the borders. The presence of two different pinning states in the specimen may then also provide an explanation why no simple functional dependence between the activation barrier \( U \) and the current density \( j \) could be found for this temperature interval.

Further, in this temperature regime the current density \( j(t_{s}) \) at the starting time \( t_{s} \simeq 10^{-1} \text{ s} \) decreases with increasing temperature extrapolating to \( \hat{T} = 0.988 \ T_{c} \). This temperature \( \hat{T} \) agrees with the low field extrapolation of the temperature \( T_{WPT}(H) \) associated with a rapid drop in \( j_{c} \).
Figure 5.18: Low field $H$-$T$/$T_c$ diagram of 2H-NbSe$_2$ ($H_{dc} \perp ab$-planes) in the neighbourhood of the critical temperature $T_c$. Lines denote: $H_{DT}(T)$ onset of the peak effect regime, $H_{PE}(T)$ estimated position of the peak in $j_c$, $H_{WPT}(T)$ maximum slope of the strong decrease of $j_c$, $H_{IL}(T)$ irreversibility line, $H_{c2}(T)$ upper critical field and $H_p(T)$ field of first flux penetration. The shaded areas indicate the different vortex states. The demagnetization factor $D \approx 0.63$ has been neglected.
6 Overview and Future Prospects

In the first part of this thesis, we investigated the whole low field $H$-$T$ diagram of the high temperature superconductor $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8_{+\delta}$ with the magnetic field applied perpendicular to the superconducting $\text{CuO}_2$ layers. $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8_{+\delta}$ shows the relevant characteristics associated with the properties of HTSC's: high anisotropy, high critical temperature, and weak pinning. The good quality of the investigated single crystal and the high sensitivity of our experimental arrangement allowed us to reconstruct on the same sample the very complicate vortex matter phase diagram as compiled from several experiments on different specimens in the last decade. Moreover, in this thesis we show that the low field $H$-$T$ diagram of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8_{+\delta}$ is even more complex than previously known and apparently displays additional new vortex matter phases.

In the second part of this work, the low field behaviour of the peak effect has been investigated in the low temperature superconductor $2\text{H}-\text{NbSe}_2$. The anisotropic superconductor niobium diselenide $2\text{H}-\text{NbSe}_2$ has attracted a great deal of attention largely because many of its properties lie in between those of conventional superconductors and the high temperature cuprates. Moreover, a reentrant behaviour of the peak effect temperature which has been related to the theoretical predicted existence of a vortex liquid phase at low fields [25, 27], has been recently reported in this material [16, 17]. Our results confirm this reentrant behaviour of the peak effect temperature. However, according to our measurements, the peak effect phenomenon is not related to a melting transition but rather to an order-disorder transition as proposed very recently [18]. In addition, a new behaviour of the peak
effect temperature is observed at fields $H \gtrsim H_{cl}(T)$. Therefore, a novel low field vortex matter diagram in the neighbourhood of $T_c$ is presented.

We would like to conclude this thesis with a brief list of experiments which form a natural continuation of this work.

- Susceptibility and magnetization field sweep measurements on $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ single crystals should be performed at different cycling rates $dH/dt$ in the field regime where the new crossover $H_{13D}(T)$ presented in this thesis, has been observed. If this crossover becomes more evident for fast cycling rates $dH/dt$, it would be a further indication that this transition corresponds to a crossover from a single vortex pinning regime $[(0-1)D$ and $(1D)]$ to a vortex bundle pinning regime $[(0-3)D$ and $(3D)]$ as proposed by [12-14]. The creep rate in the $[(0-1)D$ and $(1D)]$ is predicted to be stronger than in the $[(0-3)D$ and $(3D)]$ regime, so that the steep decrease of the critical current density at the crossover should become experimentally more evident for fast cycling rates $dH/dt$.

- It would be interesting to investigate the narrow low field vortex liquid phase predicted theoretically slightly above the lower critical field $H_{cl}(T)$ [25, 27, 58], by susceptibility and magnetization field sweep measurements performed at very low cycling rates $dH/dt$ on ellipsoidal shaped $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ single crystals. This measuring procedure on a sample with this geometry remove spurious effects due to metastable configurations (Bean-Livingston barriers, geometrical barriers) [2] and consequently, the narrow low field vortex liquid phase slightly above $H_{cl}(T)$ should not be obscured anymore by surface barriers for vortex entry.

- Low field magnetization temperature sweep measurements should be performed on $2H$-$\text{NbSe}_2$ single crystals with varying amounts of quenched disorder. These investigations would elucidate the effects of quenched disorder on the peak effect transition in the field regime $H \lesssim 85$ Oe where the peak effect temperature has been detected to increase with decreasing field.
- The time relaxation of $M_{rem}$ in a 2H-NbSe$_2$ crystal should be measured at reduced temperatures $T/T_c \lesssim 0.5$. As shown in this thesis, in the regime $0.5 \lesssim T/T_c \lesssim 0.8$ a decrease of the initial decay rate with increasing temperature occurs. Accordingly, in the temperature regime $T/T_c \lesssim 0.5$ a maximum in the decay rate should be observed. This behaviour of the decay rate would then be similar to the behaviour of the creep rate detected at intermediate temperatures in YBa$_2$Cu$_3$O$_7$ single crystals [110]. In this material, the maximum in the creep rate has been interpreted as an indication of a dimensional crossover from a single vortex line pinning (1D-pinning) to a vortex bundle pinning (3D-pinning) regime predicted for continuous anisotropic superconductors by the weak collective pinning theory [110].
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Appendix A AC-Susceptibility

The measurement of complex susceptibility $\chi = \chi' - i\chi''$ in a weak ac-field, is one of the most useful methods for the study of magnetic response of superconductors. Superimposing a small ac-field $H(\omega t) = H_{ac}\text{Re}(\exp(i\omega t))$ on a large dc-field $H_{dc}$, the resulting time-dependent magnetization $M(\omega t)$ of a specimen can be expressed in the form of the Fourier expansion [102]

$$M(\omega t) = \chi_0 H_{dc} + H_{ac} \sum_{n \geq 1, n \in N} \text{Re}(\chi_n \exp(i\omega t))$$  \hspace{1cm} (A.1)

where $\text{Re}()$ denotes the real part of the complex variable and $\omega$ is the fundamental frequency. The first term is a time independent contribution or 'offset' which is due to the presence of the dc-field $H_{dc}$. The second term is the time dependent component associated with the ac-field $H_{ac}\cos(\omega t)$. The harmonic susceptibility $\chi_n = \chi'_n - i\chi''_n$ are the Fourier coefficient of the magnetization, where $\chi_1$ is the fundamental susceptibility and the others are the higher-harmonic susceptibilities. Experimentally one usually determines the real part $\chi'_1$ and the imaginary part $\chi''_1$ of the susceptibility by measuring change of inductance and effective resistance of a pickup coil surrounding the sample. For facility, in the following we will write $\chi_1 = \chi$. 

119
There have been mainly three types of models suggested in literature in various microscopic physical pictures for the susceptibility of non ideal type-II superconductors [102].

The first type of model assumes a temperature (and field) dependent relaxation time $\tau$, which measures how fast the system approaches equilibrium after a disturbance [132–134]. The resulting expression for the complex susceptibility is usually of the Debye form $\chi = \chi_{o}/(1 + i\omega\tau) = \chi' - i\chi''$ where $\chi_{o}$ is the static susceptibility and $\omega$ is the frequency of the perturbing ac-field. It follows

$$\chi' = \frac{\chi_{o}}{1 + (\omega\tau)^2} \quad (A.2)$$

$$\chi'' = \frac{\chi_{o}\omega\tau}{1 + (\omega\tau)^2} \quad (A.3)$$

In this model, a $\chi''$-peak results at the temperature at which $1/\tau$ reaches the measurements frequency $\omega$.

The second type of model emphasizes the diffusive motion of flux lines [103]. When a superconductor shows a linear resistivity $\rho$ due to flux motion, regardless of its origins, the penetration depth of the ac-field is a skin-depth $\delta_{s} = (c^{2}\rho/2\pi\omega)^{1/2}$. The real and imaginary parts of susceptibility in the case of a slab geometry have the following forms

$$4\pi\chi' = -1 + \frac{\sinh(u) + \sin(u)}{u(cosh(u) + cos(u))} \quad (A.4)$$

$$4\pi\chi'' = \frac{\sinh(u) - \sin(u)}{u(cosh(u) + cos(u))} \quad (A.5)$$

where $u = d/\delta_{s}$ with $d$ the sample thickness. The imaginary part $\chi''$ reaches a maximum at $\omega_{peak} \simeq 0.8c^{2}\rho[H,T]/d^{2}$, where $c$ is the speed of light.

The third model is the nonlinear response critical state model [123, 124], in which the susceptibility results from hysteretic penetration of magnetic fluxoids. The expression for the real and the imaginary part of $\chi$ are the following [102]
Appendix A. AC-Susceptibility

\[ 4\pi \chi' = -1 + \frac{H_{ac}}{4\pi j_{c}d}, \quad H_{ac} < H^*, \quad (A.6) \]

\[ \chi'' = \frac{H_{ac}}{3\pi^2 j_{c}d}, \quad H_{ac} < H^* \quad (A.7) \]

\[ 4\pi \chi' = -\frac{\pi j_{c}d}{H_{ac}}, \quad H_{ac} > H^*, \quad (A.8) \]

\[ 4\pi \chi'' = \frac{4j_{c}d}{H_{ac}} - \frac{16\pi j_{c}^2 d^2}{3H_{ac}}, \quad H_{ac} > H^*. \quad (A.9) \]

where \( j_{c} \) is the critical current, \( d \) is the sample dimension and \( H^* \) defines the minimal field in the Bean model where \( B \neq 0 \) over the entire sample. Equation A.8 and A.9 are only numerical approximation.

The major differences among the three models presented above are that the first two are independent on the amplitude \( H_{ac} \) (linear response) but dependent on the frequency of the ac-field while the critical state model does depend on the amplitude of the ac-field (nonlinear-response) and could be frequency dependent if the critical current density is time dependent.

These three models have been compared with susceptibility measurements performed on a conventional superconductor Nb_3Al and on an unconventional superconductor YBa_2Cu_3O_{7-\delta} by Ling and Budnick [102]. Their conclusions are the followings: The first model is not appropriate to describe superconductors. At low ac-fields applied parallel to the dc-field, a superconductor with pinning can give linear response signal and can be better described by the diffusive model. The critical state model is instead more relevant for zero dc-fields, ac-field applied normal to the dc-field or ac-field applied parallel to the dc-field when the amplitude is large. According to [102] and due to the fact, that our susceptibility measurements performed on the Bi_2Sr_2CaCu_2O_{8+\delta} single crystal did not show any dependence on the amplitude \( H_{ac} \) (linear response) of the ac-field (see figure A.1), the susceptibility data obtained on the BSCCO sample have been described within the diffusive model.
Figure A.1: $\chi'(H)$ and $\chi''(H)$ data performed at a temperature $T = 45 \text{ K}$ on a $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ single crystal with an ac-field of frequency $\omega = 160 \text{ Hz}$ and different amplitudes $H_{ac}$.
Appendix B  Bean Model for Strips and Disks

In the case of an infinite slab parallel to the applied magnetic field \( H \), the dependence between \( M \) and \( j \) has been described by Bean [123,124]. Gurevich and Brandt [135] showed that despite the particular field distribution for strips and disks sample geometries, the current density \( j \) can still be considered as constant throughout the sample at a given time \( t \). It follows that the magnetization \( M \), which is given by [136]

\[
M(t) = \frac{1}{V} \cdot \frac{1}{2c} \int r \wedge j(r,t) \, dV, \tag{B.1}
\]

for a disk-like geometry, for a constant current density \( j(r,t) = j(t) e_\phi \), can be expressed as

\[
|M(t)| = j(t) \cdot \frac{1}{V} \cdot \frac{1}{2c} \int |r \wedge e_\phi| \, dV, \tag{B.2}
\]

where the integration over the geometrical factor leads to

\[
M(t) \simeq \frac{R}{3c} \cdot j(t), \tag{B.3}
\]

with \( R \) being the sample radius. For the case of disks (strips) the well known Bean model relationship for an infinite cylinder (infinite slab) in the fully critical state is therefore still a valid approximation.
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List of Publications

- M. Nideröst, R. Frassanito, M. Saalfrank, A.C. Mota, G. Blatter, V. N. Zavaritsky, T.W. Li and P.H. Kes
  "Lower Critical Field $H_{c1}$ and Barriers for Vortex Entry in Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ Crystals".

- M. Saalfrank, M. Nideröst, A.C. Mota, and P.H. Kes
  "Magnetization Studies in 2H-NbSe$_2$ Single Crystals at Very Low Fields Near $T_c$".

- M. Saalfrank, M. Nideröst, A.C. Mota, G. Blatter, T.W. Li and P.H. Kes
  to be published
  "Vortex Diagram in Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$: Possible New Low Field Vortex Matter Phases".

- M. Saalfrank, M. Nideröst, A.C. Mota, and G. Blatter
  to be published
  "Magnetic Studies of the Peak Effect in the Anisotropic Superconductor 2H-NbSe$_2$ at Very Low Magnetic Fields".