Apparatus and Instrumentation Design for Investigation of Surface Impact Effects on Superconductivity

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APPARATUS AND INSTRUMENTATION DESIGN FOR INVESTIGATION OF SURFACE IMPACT EFFECTS ON SUPERCONDUCTIVITY

A Thesis
Presented to
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In Partial Fulfillment
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Abstract

The effects of ion irradiation on the physical properties of materials make EBITs an invaluable tool for many scientific and engineering fields. Many experiments rely on the use of these lab setups to test for device reliability, explore surface physics phenomena, and replicate the environment for many physical systems that are not readily accessible. We seek to extend the capabilities of these experiments using the CUEBIT and a new sample holder installed in section 3.

This thesis begins by presenting an overview of the CUEBIT and the basic operations of the equipment. This is followed by a brief explanation of surface physics effects on superconductivity to provide a background into one of the experiments that are planned to be conducted in the CUEBIT with the new equipment.

Chapter 2 will provide a description of the section 3 setup and the new sample holder that was installed, as well as, its planned uses. An explanation of beam characterization using the Faraday cup and faceplate is also provided in this chapter.

Chapter 3 shows simulated and calculated results for the setup. Examples are given of data using the niobium experiment that is described in chapter 1. An introduction to a new plotting program, SRIMP, is also given as well as initial results from the software.

The thesis concludes with preliminary data for the section 3 sample holder setup. Plots of beam current on both the Faraday cup and faceplate are given. This is all concluded by a look at two future research projects planned for section 3 and concluding remarks.
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Chapter 1

Introduction

1.1 The EBIT

The primary piece of equipment used in this experiment was the Clemson University Electron Beam Ion Trap (CUEBIT). The CUEBIT can operate as either an ion trap (EBIT) or source (EBIS). For the purpose of this experiment, we are concerned with its EBIS properties for use in target irradiation.

The CUEBIT operates by first powering a cathode that can produce a current of up to several hundred mA. An anode in front of the cathode is given a large positive potential which strips electrons off the cathode and into the drift tubes. A large, 6T, superconducting magnetic compresses the extracted electrons down into a narrow beam. This electron beam floating down the drift tube serves two purposes. First, it ionizes the gas in the drift tubes, and second, it functions to keep the ions trapped radially around the beam. Two electrodes at either end of the drift tubes are given a positive voltage which traps the ions in the axial directions.

The electrodes and electron beam work together to provide the CUEBIT with its ion-trapping functionality. In order to use the CUEBIT as an EBIS, we must lower the potential on the electrode that faces the extraction beamline. This allows for the ions to leak out and be used in experiments further down the beamline. The endcap can also be cycled on and off allowing for pulses of ions, however, for the purpose of this experiment, we are only concerned with the leaky operation of the extraction beamline.

After the endcap sits an electron collector to stop any electrons that have made it from the
Figure 1.1: Diagram of the CUEBIT used for this experiment. All data were collected in section 3
cathode from entering the extraction beamline. An extraction lens will then focus the ions down into a more confined beam and a set of einzel lenses and deflectors will move the ions from the trap down toward the variable strength electromagnetic at section 7 in figure 1.1. Since this magnetic is designed with a $90^\circ$ turn, by varying the magnetic field strength of this analyzing magnetic we are able to select ions with only specific charge-to-mass ratios to continue down the beamline.

EBIT systems offer multiple advantages over other forms of ion beam creation. First, particles at the energies we are attempting to use would require either a pressurized source or a hot aperture designed to produce a desired energy distribution. This would be quite difficult in higher-energy regimes since the needed pressures would become difficult to achieve while also maintaining a stable chamber pressure. The hot aperture setup would fail due to the fact that it would begin to ionize other particles above certain temperatures. We could also not use a neutral beam, since that would mean we could no longer easily focus and transport the particles. Finally, neutral particles are also unable to be accelerated by any physical means.

All of these issues are not a factor in the CUEBIT since it utilizes an ionizing beam source and the previously mentioned charged-particle optical setup. All the previously mentioned components are pictured in figure 1.1.

### 1.2 Surface Physics and Superconductivity

#### 1.2.1 Ion Irradiation

The purpose of this experiment is to utilize the CUEBIT to irradiate target niobium films with ions to analyze the effects this has on the superconducting properties of the films. To do so, we must first look at the effects of ion irradiation on material surfaces.

When analyzing the effects of ion irradiation on a material, we must consider if we are working with singly or multi-charged ions. This becomes important as previous results have found that singly charged ion irradiation is generally dominated by nuclei-nuclei interactions[10]. The electron in these singly charged ions mainly serves to act as a screen for the ion’s nuclear charge.

We begin to see more interesting effects take place when we look at multi-charged ion irradiation. This is because multi-charged ions will have a greater desorption effect than that of singly charged ions[4]. These multi-charged ions are also of interest because the summed binding energies, or reneutralization energy, of these ions, are comparable to that of the kinetic energy of
the ions themselves. This renuetralization energy would cause material modifications very similar to that of direct ion impact.

These differences we see in the interactions of singly versus multi-charged ions lead to changes in how the material is damaged by the beam. For singly charged ion beams, we usually see something similar to a track formation[1] while with multi-charged ions, we see that the ion will dissipate its energy in a nano-meter-sized volume when it interacts with target material[2]. This allows for multi-charged ions to be more controlled in where and how the material is damaged compared to singly charged ions.

For the purposes of this work we are not concerned with the location and shape of the ion damage and so multi-charged ions will be used in the irradiation process. This is because the CUEBIT supports the production of multi-charged ions much better than singly charged and because multi-charged ions will produce more damage per impact than the singly charged.

1.2.2 Impurities and Superconductivity

In this experiment, we are concerned with the effect of ion irradiation on the superconducting properties of niobium, specifically its critical temperature. We should begin by looking at what expected results the irradiation would have on the niobium and what this should mean for its critical temperature.

The two major effects on resistivity that we should consider are impurities in the crystal and phonons. Both impurities and phonons will increase resistivity since they are breaking the crystalline structure of their system. The phonon issue is solved by decreasing the temperature of the system until the vibration of nuclei is low enough to support superconductivity.

In order to understand the effect of impurities on critical temperature we can use BCS[3] theory to construct the equation

\[ kT_c = 1.14E_D * e^{1/N(0)V} \]  

(1.1)

where \( E_D \) is the debye cutoff energy, \( N(0) \) is the electron density of states at the Fermi level, and \( V \) is electron-phonon coupling potential. We can see that the parameters on the right-hand side are going to be dependent on the impurities within the crystal. Equation 1 confirms that adding impurities to the crystal structure will result in a decrease in \( T_c \) as we had hoped for the purposes
of the study.

It should be noted that the equation and treatment of impurities in a superconductor as treated in the previous paragraph with BCS theory is not a fully complete model as it does give rise to some issues in certain extremes of impurity density[8]. However, the full treatment only changes the shape of the $T_c$ decreases as seen in figure 1.2, but the decrease still persists as hoped so for the purposes of this experiment the BCS treatment is satisfactory.

Combining the previous BCS theory with the ion irradiation discussed in 1.2.1, we can begin to see the motivation for our niobium experiment. We know that the niobium will be irradiated with multi-charged ions and thus will create impurities in the structure of the niobium. These impurities should then change the $T_c$ of the niobium from its original value. The purpose of this experiment is to analyze the correlation, if any exist, between irradiation dosage and $T_c$ changes.

### 1.2.3 Experimental Overview

Now that we have discussed the basics of ion irradiation, and we have also discussed the effects of impurities on $T_c$, a brief description and motivation of the experiment are given with a
more detailed description to follow in chapter 2 with simulated data to be discussed in chapter 3 and current data from the CUEBIT presented in chapter 4.

The setup for this experiment involves thin niobium films seated on silicon that will be inserted into section 3 of the CUEBIT and irradiated with varying doses. $T_c$ measurements of the samples would be taken before any irradiation and after irradiation. $T_c$ measurements will be taken and analysis will be conducted between irradiation dosage and $T_c$ changes.

In order to perform this experiment, a new mag-arm was fitted onto section 3 of the CUEBIT. This new arm has the ability to rotate 360° and the end is fitted with a metal cubic sample holder that is capable of being loaded with up to three samples simultaneously with the fourth side of the cube reserved for a faraday cup. A full description and figures of the section 3 arm are contained in section 2.2.

The goal of the experiment is to load the section 3 arm with three niobium samples to be irradiated. Once the samples are loaded and the vacuum is restored to section 3, the faraday cup would be rotated into the beamline. The faraday cup will serve two purposes, first, because the cup is fitted with a faceplate, it can be used to align the beamline onto the sample holder. Second, the cup allows for current measurements to be made that are used for dosage calculations in section 3.1. After measurements are made with the faraday cup, one of the niobium samples would then be rotated into the beamline and the irradiation would begin. Because samples can be easily rotated in and out of the beam path, we can give samples different dosages by changing the time in which they are exposed to the beam. For this experiment, each sample would be exposed for a different length of time resulting in different dosages on each.

Once all samples have been irradiated, $T_c$ measurements would be conducted on the samples to see if and how much change there was in the $T_c$ on the niobium and if that change has any correlation to the irradiation dosages.
Chapter 2

Experimental Design and Methods

2.1 Introduction

In order to facilitate easier irradiation of multiple samples and in particular to allow our niobium samples to be quickly interchanged for different radiation dosages, section 3 of the CUEBIT was fitted with a new rotatable mag arm. This new arm provided two benefits over previous designs because 1) it was fitted with a Faraday cup/faceplate on one side which allows us to analyze the beam density and 2) it can be rotated through 4 faces which allow for irradiation of multiple samples without the need to stop the beam or pump down section 3.

The sample holder mentioned above was designed and fabricated by the departmental machine shop. The following chapter provides an overview of the experimental setup for the niobium \( T_c \) experiment. This will include a summary of the section 3 arm design and its implementation, and an explanation of how the CUEBIT will be involved, and concludes with a brief description of the Faraday cup and faceplate setup on the section 3 arm.

2.2 Section 3 Arm

For this experiment, section 3 of the CUEBIT was fitted with a new sample holder pictured in figure 2.1. This holder involves a metal cube situated onto an adjustable mag rod. Three sides of the cube are fitted with slots to hold sample platters and the fourth side was designed to hold a Faraday cup and a thin metal plate with a small hole was screwed atop the cup. The thin metal
plate is what is referred to as the faceplate. The purpose of the faceplate and Faraday cup setup will be explained in more depth in section 2.3. As previously mentioned, the sample holder is mounted onto an adjustable mag rod. The mag rod involves a magnetic inner rod resting on ball bearings placed inside a larger metal rod. The outer rod is fitted with a strong magnet that can be moved up and down the arm, as well as, rotated around the arm. The magnet outside the arm is paired with the magnetic rod inside which allows for the rod to be manipulated from the outside. The mag rod allows the sample holder to move throughout the x-axis and rotation 360° about the mag rod.

The Faraday cup/faceplate setup requires separate wired connections to both the cup and faceplate. This was achieved by first fitting one wire to the screw going through the platen that the Faraday cup is resting on. This screw can be seen going through the platen in figure 2.1. Since the faceplate needs to be electrically disconnected from the Faraday cup, a layer of Kapton was placed between the faceplate and the cup. Since the screws holding the faceplate to the holder are also connected to the sample holder, the screws are also covered in Kapton tape making the faceplate completely isolated from the cup. The faceplate wire is connected under one of the faceplate screw heads and tightened against the faceplate. The need for these components to be isolated will be explained more completely in section 2.3.

Figure 2.1: CAD model of section 3 mag arm
2.3 Experimental Setup

The general setup for experiments in section 3 goes as follows. The desired beam of ions is created in CUEBIT using the process outlined in section 1.1. After the ions have been created they are aligned in section 3 using the Faraday cup. After the beamline has been centered on the sample holder we can measure the current of the beamline and from this, we can calculate the ion flux. After the flux is measured, we can begin rotating samples into the beamline. Once the sample is rotated into position, we can then determine the irradiation time by dividing the desired dosage by the measured flux. Since the three samples can be freely rotated we can provide different dosages to the samples.

2.4 Faraday Cup and Deconvolution

Previous techniques have been developed for creating profiles of beam current density[9]. In order for this process to be used the Faraday cup and faceplate setup that is described in section 2.2 is required. A description of this process is presented below.

In order to create a profile of the current density, \( j(r) \), a large grid of current readings is taken and each point on one of these grids at location \( r \) can be represented as a convolution of the Faraday cup detector function, \( d(r) \), with the current density at \( r \). If we wish to obtain the full function for current density we must deconvolve the detector function out.

We begin by representing the current by

\[
I(r) = \int d(r - r')j(r')dr'
\]

(2.1)

the detector function is written as

\[
d(r) = \begin{cases} 
1 & \text{if } r \leq R \\
0 & \text{if } r > R 
\end{cases}
\]

(2.2)

where \( R \) is the radius of the Faraday cup. By linearly interpolating between points in the measured grid we obtain a square grid with 41 points. This linearly interpolated grid is padded with zeros to form a larger grid of 137 points on a side. The padding is performed to avoid the creation of artifacts by the processes of convolution and deconvolution. Because we have finely spaced grids that
fall to zero outside of a finite-sized region, we are able to use continuous and finite-length discrete Fourier transforms interchangeably. Therefore, using a continuous Fourier transform we obtain the detector function as

\[ D(k) = \int d(r)e^{ik \cdot r}dr \]  
(2.3)

a finite-length discrete Fourier series of I(k) can be written as

\[ I(k) = D(k)j(k) \]  
(2.4)

we then define two new quantities \( I_n(k) = I(k)/D(0) \) and \( D_n(k) = D(k)/D(0) \) so equation 2.4 is written as

\[ I_n(k) = D_n(k)j(k) \]  
(2.5)

For our purposes \( D(0) = 2\pi R^2 \). When we take the Fourier transform of the detector function and divide by \( \pi R^2 \), we obtain the function \( D_n(k) = 2J_1(kR)/kR \) where \( J_1 \) is a Bessel function of the first kind of order one and \( k = |k| \). After this, we need to eliminate noise from the deconvolution using Wiener optimization[6].

We can write the current of as a combination of the signal, \( s(r) \), and noise, \( n(r) \).

\[ I(r) = s(r) + n(r) = d(r) \otimes j(r) + n(r) \]  
(2.6)

note that in this equation, \( \otimes \) is the convolution. We now define the noise-to-signal power ratio as

\[ \eta_n(k) = \frac{1}{|D(0)|^2} \frac{|N(k)|^2}{|J(k)|^2} \]  
(2.7)

where \( N(k) \) is the Fourier transform of the noise. We can then estimate the current density using Weiner optimization as

\[ \tilde{J}(k) = \frac{I_n(k)D_n(k)*}{|D_n(k)|^2 + \eta_n(k)} \]  
(2.8)

This means that given the approximation and \( \eta_n(k) \), we can find the current density by
taking the inverse Fourier transform of equation 2.8.
Chapter 3

Simulated Results

In this chapter, the simulated and calculated damage of the ion beam on the niobium samples is discussed. As well, in order to allow for more efficient graphing of the simulated penetration depths, a new program was created that interfaces with the ion penetration simulating software SRIM (The Stopping Range of Ions in Matter). SRIM allows for the input of an ion beam and sample with various parameters and it will then simulate penetration depth. For the purposes of this experiment, we wished to create a graph of penetration depth vs ion energy to determine the required beam energy for the experiment. In order to automate this process, a new program, SRIMP (The Stopping Range of Ions in Matter Plotter), was created to graph just this. A full description of the program and example plots will be presented in section 3.2.

3.1 Ion Flux and Surface Removal

In order to determine the best energy to irradiate the niobium sample with, we need to be able to calculate the flux and surface removal of the beam so we can find the proper energy that will cause adequate damage to the sample to change the $T_c$. However, we do not want to cause too much damage that the sample loses most of its superconducting properties. The following two sections will outline how the flux and damage are calculated and the final section will conclude with some results using a sample beam and literature values.
3.1.1 Ion Flux

Because of the Faraday cup in section 3, we are able to read the current for the beamline and we also know the charge state of the ion from the bending magnet discussed in section 1.1. This makes it very easy for us to determine the flux of ions arriving at section 3 by starting with the definition of current

\[ I = \frac{q_{\text{tot}}}{t} \]  

(3.1)

where \( q_{\text{tot}} \) is the total charge passing through the beam area in time, we can replace \( q_{\text{tot}} \) with the expression \( n_i \ast n \ast e \) where \( n_i \) is the number of ions, \( n \) is the ion charge number, and \( e \) is the elementary charge. By making this substitution into equation 3.1 and rearranging we can get an expression for the number of ions that pass through the beam area in time \( t \).

\[ n_i(t) = \frac{It}{ne} \]  

(3.2)

3.1.2 Layer Removal

For this experiment, it was decided that the beam area would be larger than the sample surface area. We are also assuming a uniform distribution of ions within the beam. We can express the ratio of ions from the beam that hit the sample as \( \frac{A_s}{A_b} \) where \( A_s \) is the surface area of the sample, and \( A_b \) is the beam area. If we multiply equation 3.2 by this ratio and the sputter yield (S) we can get an expression for the number of atoms sputtered in time \( t \) (\( n_s(t) \))

\[ n_s(t) = \frac{ISA_s t}{neA_b} \]  

(3.3)

Since Niobium is a BCC lattice we need to analyze two different layers when finding the number of atoms per layer. First, we have what we will refer to as the ”cubic layer” which is the layer made from the basic cubic lattice structure (blue spheres in figure 3.1) and another layer made up of the center atoms (red sphere) that we will call the ”center layer”.

We can then say that a cubic layer has \( (c_w+1)(c_l+1) \) atoms per layer, where \( c_l \) and \( c_w \) are the numbers of cells in the length and width respectively. For the center layer, this expression becomes just \( c_w \ast c_l \).
Since we know $c_l$ and $c_w$ are very large numbers we can approximate $(c_w+1)(c_l+1)$ to simply be $(c_l)(c_w)$. This means that both layers will have approximately the same number of atoms ($n_l$) and we can express this as

$$n_l = c_l \times c_w = c = \frac{A_s}{a^2} \tag{3.4}$$

where $c$ is the number of cells per layer, and $a$ is the lattice constant.

Now that we know the number of atoms per layer we can get a final expression for the number of layers removed as a function of time ($L_r(t)$) by diving equation 3 by equation 4.

$$L_r(t) = \frac{ISA^2t}{ne\pi r_b^2} \tag{3.5}$$

where $r_b$ is the radius of the beam.

One important note is that the layers removed are independent of the surface area of the sample since we are using a beam that is larger than the sample.

### 3.1.3 Simulation Results with Literature Numbers

Now that we have a function for Layer removal, we can plot expected layer and depth removal over time given the sputter yield. The current literature values for argon and niobium sputter yields are inconclusive so a plot was created using sputter yields of 1, 0.6[5], and 3.4[11]. This plot is shown in figure 3.2 with both layers and depth removal labeled.
3.2 SRIMP

The final simulations done for this project required the use of SRIM to create plots of ion range vs. energy. This allowed us to determine the most likely penetration depths and best ion energies to use in the experiment. Since SRIM in its current implementation, does not produce plots of range and straggling, it was required to create a new program, SRIMP, that interfaces with SRIM and graphs the simulated data. Example plots of SRIMP containing results for neon and niobium simulations are shown in figure 3.3.

3.2.1 SRIMP Design

SRIM allows for external programs to interface with it via the SR module folder. This is a subfolder within SRIM that contains an executable that provides the same simulations as SRIM but without the GUI. Parameters for the simulation are inputted by manipulating the SR.in file within the SR module folder. This file contains all the same parameters as the GUI version. The resulting
simulation data is then stored in a text file with a name specified by the user.

Creating a program to interface with SRIM is fairly trivial, as the program only needs to contain a function to overwrite the SR.in file with the user-specified parameters and a second function that creates a process to run the SRIM executable. The program then waits for the SRIM process to finish and reads the data in the resulting text file. Finally, a plot is created from the data using whatever plotting library is best for the language being used, in our case jfree chart was used to create plots in java. The plots contain the range vs. energy with error bars created using the straggling values.

After the initial program was created, a GUI was added to allow for SRIMP to be used as easily as SRIM. The GUI requests all the same parameters as SRIM, and the user can specify plot units and energy ranges. The source code and jar file for SRIMP can be found at (https://github.com/TarjeTart/SRIMplots) along with instructions on its use.
Chapter 4

Results

In this chapter, we will review the current data that has been collected in section 3 with the new arm and sample holder. At the time of writing this, the only data from the new setup is current readings from the Faraday cup for testing that the cup and faceplate were reading correctly.

4.1 Section 3 Faraday Cup Test

Following cathode issues and changes in the CUEBIT source, pressures were able to be achieved that allowed for ion beams to be created with the background gas. These ions were focused into section 3 and current measurements were taken on both the faceplate and Faraday cup. To test if the faceplate and cup were working properly, multiple runs of current were taken with the beam deflected onto and off of the sample holder. The background deflected and undeflected data was plotted in golang, along with normal curves of the data to confirm that the current readings with the ion beam focused onto the arm was statistically significant from the background noise.

Some samples of the plots are shown below. The full set of graphs can be found in the appendix.
Figure 4.1: Plot of current over time of deflected (blue) and undeflected (green) current readings for Faraday cup

Figure 4.2: Plot of current over time of deflected (blue) and undeflected (green) current readings for faceplate
Chapter 5

Conclusions and Discussion

In the concluding chapter two future projects that will utilize the section 3 mag arm are briefly described. Following that will then be concluding remarks for the project.

5.1 Future Research

The first of the two future projects to utilize the section 3 arm has already been discussed in the previous chapters. The niobium irradiation experiment is expected to begin once pressures in the CUEBIT source have lowered to the point that neon or argon gas can be released into it without the pressure going too high that the cathode is no longer able to emit properly. Background gas like oxygen is not usable as an ion source since the niobium would be at risk of oxidizing which introduces new effects that are not of interest to this project. Once this point is reached the experiment should be conducted as described in chapter 2 section 3. The samples would then have $T_c$ measurements taken and compared to the measurements taken before, as well as, compared to each other with different irradiation dosages.

The second experiment that is expected to take place using the new sample holder is a project involving P3HT transistors. The goal of this project would be to irradiate the transistors with different dosages using the same process as previously mentioned. For this project, the goal is to test how well the transistors can withstand ion bombardment and continue to function properly. This experiment could be conducted using background gas ions since the P3HT should not oxidize during the irradiation.
5.2 conclusion

This thesis has been devoted to discussing changes to the section 3 arm within the CUEBIT necessary to accommodate new experiments and to allow for easier studies involving varying irradiation dosages. The background of the CUEBIT setup is discussed as well as background information for the niobium irradiation project. A full description of the changes to the section 3 arm is given with current data confirming the proper installation of the Faraday cup and faceplate. Chapter 3 also reviews simulated results and provides an introduction to the SRIMP program. The final chapter provides a brief introduction to the two projects planned for the section 3 setup.
Appendices
Appendix A  Current Plots for Faraday cup/Faceplate Testing

Figure 1: Plot of current over time of deflected (blue) and undeflected (green) current readings
Figure 2: Time averaged plot of current over time of deflected (blue) and undeflected (green) current readings

Figure 3: Normal distributions of deflected (blue) and undeflected (green) current readings
Figure 4: Plot of current over time of deflected (blue) and undeflected (green) current readings

Figure 5: Time averaged plot of current over time of deflected (blue) and undeflected (green) current readings
Figure 6: Normal distributions of deflected (blue) and undeflected (green) current readings.

Figure 7: Plot of current over time of deflected (blue) and undeflected (green) current readings.
Figure 8: Time averaged plot of current over time of deflected (blue) and undeflected (green) current readings

Figure 9: Normal distributions of deflected (blue) and undeflected (green) current readings
Figure 10: Plot of current over time of deflected (blue) and undeflected (green) current readings

Time Averaged Data (n= 10)

Figure 11: Time averaged plot of current over time of deflected (blue) and undeflected (green) current readings
Figure 12: Normal distributions of deflected (blue) and undeflected (green) current readings

Figure 13: Plot of current over time of deflected (blue) and undeflected (green) current readings
Figure 14: Time averaged plot of current over time of deflected (blue) and undeflected (green) current readings

Figure 15: Normal distributions of deflected (blue) and undeflected (green) current readings
Figure 16: Plot of current over time of deflected (blue) and undeflected (green) current readings

Figure 17: Time averaged plot of current over time of deflected (blue) and undeflected (green) current readings
Figure 18: Normal distributions of deflected (blue) and undeflected (green) current readings
Bibliography


