Chapter 4

Radio Frequency Vortex Dynamics in MgB$_2$

The vortex dynamics of superconducting MgB$_2$ in radio frequency regime is studied. We employ the tunnel diode based technique for the measurement of shift in rf penetration depth. We find strong intergrain pinning in polycrystalline MgB$_2$. Ion irradiated films of MgB$_2$ are investigated to find reflection of post irradiation modification in band scattering mechanism using two gap BCS model.
4.1 Introduction

Starting from the early work of Gittleman and Rosenblum [1, 2], the electrodynamic response of superconductors at radio and microwave frequencies has always attracted considerable attention. More than various applications in communication related devices such as resonators and filters to the design of magnets for NMR and MRI machines, it turns out to be the most suitable frequency range to decipher the vortex dynamics properties. The first advantage is that the probe currents involved are extremely small (~0.1 mA) and secondly these techniques are generally contact less. With a superimposed DC magnetic field, the oscillating fields of rf or microwave couple to the magnetic flux lines and thus a rich variety of phenomena depending on pinning, anisotropy, vortex density and granularity can be studied. Vortex regimes that would otherwise require extremely high field with DC currents can be achieved at moderate fields with rf currents. Further, as described in chapter 2, the inductive part of the complex response of rf penetration is directly related to the penetration depth. Since the temperature dependence of rf penetration depth is related to the density of low level excitations, this parameterization yields a plethora of information regarding the symmetry of the order parameter and anisotropy of superconducting gap [3]. For example, in conventional superconductor in clean limit, where the mean free path $l$ is larger than the coherence length $\xi$, s-wave pairing mechanism is reported and penetration depth in the low temperature regime vanish exponentially as described by BCS [4]. On the other hand, in High $T_c$ compounds, the so called d-wave pairing is established from power law dependence of penetration depth as a function of temperature [5-8].

MgB$_2$ is a phonon mediated superconductor [9-12], but because of multiband properties, it is expected that the temperature dependence of penetration depth will also reflect the anisotropy in the superconducting gap.
Indeed, the superconducting state in MgB$_2$ is studied by many groups employing various techniques such as ac susceptibility, muon spin rotation ($\mu$SR), optical conductivity and small angle neutron scattering (SANS) [5-8]. Some of these reports have concluded that in the temperature range from $\sim T_c/2$ to lowest achievable temperature, $\lambda(T)$ follows $T^n$ power law dependence with $n \sim 2 - 2.7$ [5-8]. While some other reports suggest the exponential behavior [14]. So the influence of two gaps on the temperature dependence of $\lambda(T)$ is still an open question. As established in chapter 3, with irradiation induced defect it is possible to tune the intraband scattering mechanism in MgB$_2$. Study of possible modification in the superconductivity mechanism because of introduction of disorder in a multigap superconductor therefore is of great interest.

For a superconductor at constant temperature and under the absence of external magnetic field there will be no variation in rf penetration depth ($\Delta\lambda = 0$). The variation in relative penetration depth as a function of magnetic field is explained in chapter 2. As magnetic field increases from zero value, the field can penetrate only into the grain boundaries and above $H_{c1}$ of grains, the field penetrates into the superconducting grains in terms of vortices. The vortex dynamics is related to intragrain pinning. Beyond the irreversibility field, the dynamics is related to the flux flow behavior [1, 2, 15].

In this chapter we first study the behavior of variation in rf penetration depth as a function of temperature, we further study pining of vortices in terms of change in rf penetration depth with respect to varying magnetic field at various constant temperatures for the polycrystalline MgB$_2$ samples INFM (clean limit) and UW (dirty limit). From this we determine pinning force constant $k_p$ in the intergrain and intragrain regions and coefficient of viscosity in flux flow regime. We find the intergrain pinning to be stronger than the intragrain pinning. The surface electron microscopy performed on both of these samples reveals well connected clusters of grains with smoother grain boundaries for clean (INFM) sample while rough grain boundaries are evident
for relatively dirty (UW) sample. In the second part of this chapter we study the rf penetration depth behavior of MgB$_2$ in pristine and ion irradiated thin films that we used in the magnetoresistance study discussed in chapter 3. We study the variation in relative rf penetration depth as a function of temperature which fits well with exponential BCS type behavior. This confirms s-wave behavior in this compound. Post irradiation affect is seen in the modification of superconducting gap.

4.2 Experiment

Our measurements are carried out on polycrystalline and thin films sample of MgB$_2$. We note that while the rf technique is somewhat surface sensitive, in extreme type II superconductors like MgB$_2$, the problem is not so severe because London penetration depth $\lambda >>$ coherence length $\xi$. So the technique is much cleaner than STM measurements where accuracy of the order of $\xi$ is demanded. Moreover, unlike thermal conductivity or specific heat, here no contribution from phonons is measured and only magnetic and electronic excitations are probed [14]. The typical size of INFM sample is $\sim 1.1 \times 3 \times 0.4$ mm$^3$ while for UW sample size is $\sim 2.3 \times 2.4 \times 1$ mm$^3$. These samples fit into coil of LC tank circuit of tunnel diode oscillator in an orientation such that axis of coil passes through plane of maximum area of sample. Results of three thin film of MgB$_2$ comprising of unirradiated (Unirr), 200 MeV Au$^{15+}$ $1 \times 10^{12}$ ion/cm$^2$ irradiated film (Au112) and 100 MeV Si$^{8+}$ $5 \times 10^{13}$ ion/cm$^2$ irradiated film (Si513) are further mentioned. The typical size of these thin film samples is $\sim 1.1 \times 2.5$ mm$^2$ and thickness of the film is $\sim 200$ nm.
4.3 Results and discussion

4.3.1 Polycrystalline MgB₂

The polycrystalline INFM MgB₂ is a low resistance sample of resistivity $\rho(T_c) \sim 3.5 \mu\Omega\text{cm}$. The mean free path is given by $l = \frac{v_F}{\epsilon_0 \omega_p \rho(T_c)}$, with $v_F$ being Fermi velocity $\sim 4.8 \times 10^5 \text{m/sec}$ and $\omega_p$ being plasma frequency with plasma energy $\sim 5.02 \text{eV}$ [16, 17]. Therefore calculated value of the mean free path is 26.7 nm which is larger than typical value of MgB₂ coherence length $\sim 5$ nm [18]. So INFM sample is in clean limit. On the other hand resistivity of UW MgB₂ sample at transition temperature $\rho(T_c)$ is $\sim 17 \mu\Omega\text{cm}$ and it is in dirty limit. The dirtiness of UW sample is also confirmed by low residual resistivity value $\rho_{293K}/\rho_{40K}$ for UW sample which is $\sim 4$ as compare to $\sim 14$ for INFM MgB₂. The transition temperature of polycrystalline UW MgB₂ sample is 38.6 K.

4.3.1.1 Temperature dependent variation in rf penetration depth

In INFM MgB₂ sample temperature dependence of variation in rf penetration depth under the magnetic field of strength 0, 0.1, 0.5 and 1T is shown in Fig 4.1. In this data the empty coil background is subtracted and $\Delta \lambda$ on the y-axis is the variation in rf penetration depth relative to the value at 20 K. This means $\Delta \lambda$ equals to ($\lambda(T) - \lambda(20 \text{ K})$).
From the Fig 4.1 we observe that zero field inductive transition onsets at 39.7 K while the resistive transition in zero external magnetic field is 39.2 K. But the $T_c$ from resistive technique is defined at 90% of its normal state value. The variation from 2 K to 20 K was recorded and the data was fitted to 2 gap BCS analysis [4, 14] but because of negligible change in frequency as compared to the background, no clear conclusions could be derived. We also note that there is some variation of $\Delta \lambda$ in normal state as a function of magnetic field. MgB$_2$ is an intermetallic and some amount of magnetoresistance has been reported [18]. This would imply that the contribution due to normal state skin depth will be field dependent.
In Fig 4.2 zero field frequency shift is fitted with the temperature dependence of two fluid expression for $\Delta F(0, T)$ [15]. This yields $\Delta F(0, T) = (1/G)[\lambda(0, 0) [1-(1-(T/T_c)^2)^{1/2}]]$. The fitting along with the experimental data is shown in Fig 4.2 and yield $\lambda(0, 0)/G = 102$. The typical value of $\lambda(0, 0)$ for polycrystalline MgB$_2$ varies in the range of 1000 - 1500 $\mu$m [4] thus we estimate the value of $G \sim 10$-14 $\mu$m/Hz. A shoulder around 25 K is also observed which is seen in similar studies carried out by Prozorov et al. [19] and it is assigned to metallic magnesium.

![Fig 4.2: Fitting of the variation in rf penetration depth with two fluid model as a function of temperature and at zero external magnetic field.](image)

**Fig 4.2:** Fitting of the variation in rf penetration depth with two fluid model as a function of temperature and at zero external magnetic field.
4.3.1.2 Magnetic field dependent variation in rf penetration depth

Shown in Fig 4.3 is the magnetic field dependence of $\Delta \lambda (H, T)$ (normalized with calibration constant $G$) at various constant temperatures for INFM MgB$_2$ sample. Considering the behavior of $\Delta \lambda / G$ at 38.3 K and 27 K, $\Delta \lambda / G$ increases sharply as the temperature gets closer to the transition temperature. It goes to saturation at much smaller field as compared to low temperature behavior where the rise is not sharp.

![Graph showing the variation in rf penetration depth as a function of external DC magnetic field.](image)

**Fig 4.3:** The variation in rf penetration depth as a function of external DC magnetic field.

Following the criteria as suggested by Rosenblum and Gittleman, we assume linear response in a periodic pinning potential scenario. Confining to
vortex pinning regime, the Campbell penetration depth dominates and is expressed as $\lambda^2_c = B\phi_0/\mu_0 k_p$. Thus the pinning force constant $k_p$ can be determined from the plot of change in rf penetration depth as a function of magnetic field. The slope of such curve will give the value of $k_p$. Fig 4.4 shows $(\Delta\lambda/G)^2$ as a function of magnetic field at various constant temperatures. Two distinct regimes of flux pinning i.e. intergrain and intragrain pinning are observed. At higher fields flux flow regimes can also be identified. Fig 4.5 shows $(\Delta\lambda/G)^2$ variation with respect to magnetic field at 32.7 K. The criterions for determining crossover parameters, corresponding to change in behavior of vortex pinning are also shown.

![Fig 4.4: Square of change in frequency versus external magnetic field at various constant temperatures for INFM MgB$_2$ sample. The orientation of the sample is such that probe field $H_{ac}$ is aligned to least dimension. The coil is oriented such that $H_{ac}$ and $H_{dc}$ are orthogonal to each other.](image-url)
Fig 4.5: Square of the variation in the penetration depth is plotted with increasing magnetic field at 32.7 K for INFM MgB$_2$ sample. Criterion for crossover parameter is also depicted. Scaled variation in rf penetration depth is plotted as a function of normalized field in the inset of this figure.

The field corresponding to the crossover from intragrain pinning to flux flow regime is defined as $H_1^*$ which is analogous to irreversibility field $H^*$. On increasing the field further, another crossover in terms of change in slope occurs. This signifies transition from flux flow regime to normal state and corresponding field $H_{c2}$ is the upper critical field value. The scaled variation in rf penetration depth $(\Delta \lambda(H, T))/(\Delta \lambda^*(H_{c2}, T))$ at various temperatures in the range from 27 K to 38.3 K is further plotted against the external DC magnetic field normalized with the upper critical field ($H/H_{c2}$) in inset of Fig 4.5.
\( \Delta \lambda(H_{c2}, T) \) is the Y axis value corresponding to \( H_{c2} \) in the Fig 4.5. The collapsing of different curve corresponding to various temperatures on a single curve implies scaling phenomena. Such collapsing behavior has earlier been reported for organic superconductor by Sridhar et al. [20].

The upper critical field \( H_{c2} \) determined following the criterion explained in Fig 4.4 is plotted as a function of temperature for INFM MgB\(_2\) sample in Fig 4.6. Systemic increase in critical field values is observed with decrease in temperature. Fig 4.6 further depicts the resemblance between \( H_{c2} \) calculated from rf technique and resistive technique. Good matching is observed.

![Graph showing comparison of upper critical field \( H_{c2} \) as a function of temperature for INFM MgB\(_2\) sample from rf penetration depth study and resistive study.](image)

**Fig 4.6:** Comparison of upper critical field \( H_{c2} \) as a function of temperature for INFM MgB\(_2\) sample from rf penetration depth study and resistive study.

At field \( H < H_1^* \), vortices are confined to grain pinning region and pinning force constant values are calculated using Campbell type penetration in this region. The value of \( k_p \) in terms of \( G^2 \) is plotted as a function of
temperature in Fig 4.7. At still lower fields (corresponding to intergrain pinning) another slope change was clearly identified. We found the $k_p$ values in the intergrain regions an order of magnitude higher as compared to intragrain pinning. Pinning force constant in the intragrain pinning regime shows signatures of saturation as temperature is decreased.

![Graph](image)

**Fig 4.7**: Pinning force constant plotted as a function of temperature in intragrain pinning regime for INFM sample. Inset shows square of variation in rf penetration depth as a function of magnetic field. Two regimes are identified. Dominant intergrain pinning over intragrain pinning is indicated.

To further quantify the aspect of stronger intergrain pinning than the intragrain pinning, and to clarify the effect of grain boundaries next we study another polycrystalline MgB$_2$ that is in the dirty limit (UW sample). Magnetic
field dependence of square of variation in rf penetration depth $(\Delta\lambda/G)^2$ at various constant temperature for this sample is plotted in Fig 4.8. The intergrain pinning force constant, normalized with calibration factor in this sample was found to be one order of magnitude higher as compared to intergrain $k_{pi}$ of clean INFM specimen. It indicates that in dirty limit even the grain boundary pinning is improved [21]. However, in the UW sample $k_{pi}/G^2$ decreased rapidly with temperature as compared to INFM sample. For example, at $0.7T_c$ and $0.9T_c$, normalized value of pinning force constant are $4\times10^7$ and $1.2\times10^6$ whereas at same temperatures, INFM $k_{pi}$ values were found to be $2.6\times10^6$ and $6.5\times10^5$ respectively.

\begin{figure}
\centering
\includegraphics[width=0.8\textwidth]{Fig4.8.png}
\caption{Square of variation in rf penetration depth as a function of magnetic field at constant temperatures is plotted for UW MgB$_2$ sample.}
\end{figure}
Plotted in Fig 4.9 is the intragrain $k_p$ as a function of temperature for UW sample. This too suggests stronger intergrain pinning than for the intragrain pinning and confirms the results obtained for clean INFM MgB$_2$ sample. The values of pinning force constant in intragrain pinning region for the dirty sample is almost double than for the value corresponding to the clean sample.

![Graph showing $k_p/G^2$ vs Temperature](image)

**Fig 4.9:** Pinning force constant in the grain pinning region is plotted as a function of temperature for the UW MgB$_2$ sample.

We also note that using Rowell’s criteria [22] in which intergrain connectivity for transport current is correlated to difference of resistivity values at room temperature and at transition temperature $\Delta \rho (= \rho_{(293K)} - \rho_{(39K)})$, we find that INFM sample is better connected than the UW sample. Higher value of $\Delta \rho$ suggests lesser intergrain connectivity [22]. For our case in polycrystalline MgB$_2$ INFM sample, $\Delta \rho_{\text{INFM}}$ is 45.2 $\mu$Ω cm [23], while for UW polycrystalline MgB$_2$ sample $\Delta \rho_{\text{UW}}$ is 55 $\mu$Ω cm.
Thus we conclude that in as grown polycrystalline samples of MgE intergrain pinning dominates over the intragrain pinning. From clean to dirty limit, flux pinning is enhanced both in intergrain and intragrain regions. This situation is altogether different from the typical HTSC superconductor such as BSCCO 2223 [15] where due to presence of weak links, the intergrain vortex pinning region is negligible compared to intragrain pinning region.

4.3.2 Ion irradiated MgB$_2$ thin films

As mentioned in chapter 3, it is possible to alter the band scattering mechanism of a two band superconductor by heavy and light ion irradiation. Two of these MgB$_2$ films irradiated with 200 MeV Au$^{15+}$ (Au112) ion with fluence 1x10$^{12}$ and 100 MeV Si$^{8+}$ (Si513) ion with fluence 5x10$^{13}$ ions/cm$^2$ along with an unirradiated (Unirr) were studied for their magnetoresistance and magnetization measurements. These films were also studied using rf technique. Because of much smaller sample volume, measurements were difficult. Nevertheless in the following we summarize the data. The measurements are done for thin films in orientation such that c-axis of sample is parallel to probe field $H_{ac}$.

The cleanliness of the thin film MgB$_2$ sample is estimated following the criteria discussed earlier in this chapter. Comparison of mean free path $l$ with coherence length $\xi$ describes the applicability of clean limit for the superconductor. The normal state resistivity is measured by linear four probe magnetoresistance study, results of which are discussed in chapter. Calculated mean free path corresponding to these samples vary as given in Table 4.1. Considering the coherence length $\xi \sim 5$ nm for MgB$_2$ and following the condition that mean free path should be larger than the coherence length in a clean sample, it is evident from the Table 4.1 that unirradiated MgB$_2$ film...
close to clean limit and the irradiated films are in dirty limit [16]. This is understandable because ion irradiation basically incorporates more disorder into the system.

Table 4.1: Summary of the clean limit of the films studied in the present report.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Resistivity at $T_c$, $\rho(T_c)$ (in $\mu\Omega\cdot$cm)</th>
<th>Calculated mean free path (nm)</th>
<th>Status of sample ($\xi \sim 5$nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unirr</td>
<td>19.5</td>
<td>4.80</td>
<td>Dirty</td>
</tr>
<tr>
<td>Au112</td>
<td>32.4</td>
<td>2.88</td>
<td>Dirty</td>
</tr>
<tr>
<td>Si513</td>
<td>40.3</td>
<td>2.32</td>
<td>Dirty</td>
</tr>
</tbody>
</table>

4.3.2.1 Temperature dependent variation in rf penetration depth

Fig 4.10 depicts variation in rf penetration depth as a function of temperature for the three MgB$_2$ thin film samples. The empty coil corrected, normalized variation in rf penetration depth $\Delta\lambda/G$ is plotted. The onset transition temperature from rf penetration depth measurement are 35.9 K, 34.8 K and 35.1 K for Unirr, Au112 and Si513 samples respectively.
In Fig 4.10 we see that after extended defects due to Au$^{15+}$ ion irradiation, transition width has increased significantly as compared to point defects due to Si$^{8+}$ ion irradiation and the unirradiated sample. Rise in variation in rf penetration depth for Unirr sample in Fig 4.10 beyond 15 K has disappeared in irradiated samples. This broad shoulder in unirradiated sample is related to unreacted magnesium [19]. Thus we see that irradiation can also lead to more uniform annealing. Inset shows variation in normalized rf penetration depth at low temperature. We have confined ourselves to 0.5$T_c$, as was the case of Carrington et al.[4, 14]. The temperature dependent variation

Fig 4.10: Normalized relative variation in rf penetration depth for MgB$_2$ thin films. Inset shows the data as a function of reduced temperature. Probe field $H_{ac}$ is parallel to c-axis of the sample.
in rf penetration depth points to alteration in superconducting mechanism. For Si513 sample, the observed temperature dependence matches with Unirr sample only in low temperature regime but starts deviating with increase in temperature. For Au 112 sample this feature sets in at 0.18 $T_c$.

In chapter 3, we have concluded that $\sigma$ band can be made dirtier with extended defects. To see the reflection of band modification we have fitted our penetration depth data at low temperature for two gap BCS model [4]. For conventional $s$ - wave pairing, and $T < T_c/2$ the variation in penetration depth is given by [4]

$$\Delta \lambda(T) = \lambda(T) - \lambda(0) = \lambda(0)[\pi \Delta(0)/2k_B T]^{1/2}\exp(-\Delta(0)/k_B T)$$  \hspace{1cm} (4.1)

For $T < 15$ K, we find good fitting for the three films with Eq. 4.1. To extract the magnitude of two gaps from temperature dependent variation in rf penetration depth, total penetration depth $\lambda_{tot}$ can be thought of as a combination of $\lambda_{min}$ and $\lambda_{max}$ [23-25]

$$\lambda_{tot}^{-2} = \lambda_{min}^{-2} + \lambda_{max}^{-2} \hspace{1cm} T < T_c/2$$  \hspace{1cm} (4.2)

Fig 4.14 shows fitted data and experimental data corresponding to thin film samples used in study. In the temperature range $T < T_c/2$, variation in superconducting gap with temperature is limited to 10% [24]. So variation more than 10% can be attributed to the modification due to irradiation. The values of superconducting gap $\Delta(0)$ corresponding to $\pi$ and $\sigma$ superconducting gap of MgB$_2$ are summarized in Table 4.2. The $\Delta_\pi = 35.049k_B [3.02$ meV$]$ and $\Delta_\sigma = 86.8k_B [7.48$ meV$]$ values for Unirr sample are close to standard value $\sim 2.3$ meV and $\sim 7.2$ meV for $\pi$ and $\sigma$ band respectively.
Fig 4.14: Two gap BCS exponential fit for normalized $\Delta\lambda$ behavior below 15 K is shown for Unirr (upper layer), Si513 (middle layer) and Au112 (lower layer).
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Our unirradiated sample borders on clean limit, so some effect of modified band scattering is expected. For the case of Si513 sample the $\pi$ and $\sigma$ band gap values are 2.4 meV and 6.93 meV which become 2.65 meV and 6.34 meV respectively for Au112 sample. So after the irradiation both bands gets modified, but on close scrutiny we observe that modification in Si513 is different as compared to Au112. Moreover, with respect to the unirradiated sample, we observe more relative change in $\pi$ band of Si513 and $\sigma$ band of Au112. This supports our conclusions from H-T phase diagram [26].

Table 4.2: The value of $\Delta_\pi$ and $\Delta_\sigma$, obtained from 2 gap BCS fitting of $\Delta \lambda (T)$ for irradiated and unirradiated samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\pi$ gap (meV)</th>
<th>$\sigma$ gap (meV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unirr</td>
<td>3.02</td>
<td>7.48</td>
</tr>
<tr>
<td>Si513</td>
<td>2.40</td>
<td>6.93</td>
</tr>
<tr>
<td>Au112</td>
<td>2.65</td>
<td>6.34</td>
</tr>
</tbody>
</table>

4.4 Conclusion

In this chapter we studied the vortex dynamics properties at radio frequency in MgB$_2$ polycrystalline and thin film samples. Following the model proposed by Coffey and Clem, we have identified the intergrain pinning, intragrain pinning and flux flow regimes by plotting square of change in rf penetration depth as a function of dc magnetic field. We find that the intergrain pinning in MgB$_2$ to be stronger than intragrain pinning in polycrystalline samples. We also observe reflection of changes in scattering mechanism of ion irradiated films in the shift of rf penetration depth as a function of temperature.
References


