CHAPTER III

Sm-211 ADDITION

Melt textured $\text{REBa}_2\text{Cu}_3\text{O}_y$ (RE-123) has high current density ($J_c$) because of the superior microstructure and increase in the flux pinning. The microstructure contains features like 123 grains aligned in the $ab$-plane with reduced number of weak links in the direction of current flow, and with $\text{RE}_2\text{BaCuO}_3$ (RE-211) particles distributed in the 123 grains [1]. The presence of 211 particles is due to liquid phase loss, anisotropic growth rate of 123, and because the rate of dissolution of 211 in the liquid phase is not in accordance with growth rate of 123 [2]. The increase in flux pinning is due to pinning, at the crystallographic defects around the 123/211 interface. Such defects include twins, stacking faults and dislocations [3]. If excess 211 is taken in the starting material, a large amount of it will be left back after the peritectic reaction, which would contribute to increase in flux pinning. Murakami et al. [A] have observed an increase in $J_c$, for excess amount of Y-211 in the starting material of melt processed Y-123. McGinn et al. [5] observed a drop, and Jin et al. [6] reported to have seen no change in $J_c$, with increase in Y-211 concentration in Y-123 system. Lee et al. [7], gave a generalized explanation for the behaviour of $J_c$ with increasing concentrations of Y-211 phase in melt processed Y-123 system. According to them, the $J_c$ increases for low additions of Y-211 phase, and then remains independent of the added Y-211 concentration. For high concentration of Y-211, the $J_c$ drastically drops to a low value, this is because of the reduction in the matrix area. Apart from the flux
pinning property the presence of 211 particles in the microstructure also helps to
dissipate mechanical stresses, and thus increases the toughness of the material
[8,9].

The microstructural features and the $J_c$ dependence in melt
processed Sm-123 system with increasing Sm-211 concentration, is discussed in
this chapter. A systematic study on the Sm-123 system in that direction does not
exist is not reported. In the literature there exists exhaustive reports about the Y-
123 system, with increasing 211 concentration. An increase in $J_c$ for an optimum
211 concentration (30 mol%) has been reported. On similar lines, the dependence
of $J_c$ and other related properties is studied with an objective to observe those
properties with increasing Sm-211 concentration in melt processed Sm-123
samples. The samples were prepared in reduced oxygen atmosphere and using the
Solid Liquid Melt Growth (SLMG) route [10]. The reason for preparing in reduced
oxygen atmosphere is to suppress the formation of solid-solution which leads to
low-$T_c$ compounds, as discussed in the introduction of this thesis. It is reported that
by preparing samples in such a reduced atmosphere the solid-solution formation
can be reduced and the resulting compounds will have $T_c$ greater than 90 K [11]. In
the SLMG route the precursors used are solid 211 plus liquid phases instead of the
123 phase, and the usual melt texturing schedule is followed. After the peritectic
melting, the 211 and the liquid phases are combined to form the 123 phase below
the peritectic temperature.

The samples were prepared with increasing concentrations of Sm-211
phase, i.e., from 0 to 40 mol %, in the starting material. Melt processing was done
as discussed in the experimental chapter. The cooling rates adopted while melt texturing were 1°C/hr. The results of these are discussed below.

3.1 PRELIMINARY CHARACTERISATION

The X-ray patterns of all the samples with increasing Sm-211 concentration is shown in Fig. 3.1. These are indexed to the orthorhombic Sm-123 phase, with varying amounts of Sm-211 [12]. The lattice parameters obtained from the patterns closely matched those reported for the Sm-123 [12]. A decrease in the b and c parameter values, with an increase in the a parameter, characteristic of the low-T_c solid-solution [13] was not noted. Fig. 3.2 shows the dc electrical resistivity measurements of the samples plotted as a function of temperature. The T_c values are above 90 K confirming the minimization of solid-solution formation.

3.2 MICROSTRUCTURAL FEATURES

All the samples had large quasi-single crystalline domains of Sm-123, whose size was observed to decrease, and the number to increase, with the increase in Sm-211 concentration. On an average, the stoichiometric sample had about two domains, the 10 mol % sample had three and the 20, 30, 40 mol % samples had about 9, 15, 25 domains respectively. This happens because, the samples were processed by a non-directional melt growth technique, in which multiple nucleation of Sm-123 takes place. The above observation suggests that an important factor controlling the nucleation of Sm-123 is the concentration of Sm-211 in the starting material.
Fig. 3.1 : X-ray diffraction patterns of the samples with Sm-211 concentration of 10, 20, 30 and mol % in the starting material. Peaks corresponding to the solid-solution formation were not observed.
Fig. 3.2: dc electrical resistivity measurements of the samples as a function of temperature. The $T_c$ of the samples are above 90 K.
Fig. 3.3: Representative optical micrograph of the 10 mol% Sm-123 melt processed sample showing two misaligned 123 domains with 211 particles distributed on them.
Fig. 3.4: SEM micrographs of the samples with increasing Sm-211 concentration.

Sm-123 with (a) 10 mol% Sm-211, (b) 20 mol% Sm-211, (c) 30 mol% Sm-211,
(d) 40 mol% Sm-211.
Fig. 3.3 is a representative polarized optical micrograph of the sample. It shows a boundary between two domains aligned in different directions.

The microstructures of the samples (Fig. 3.4) have the usual features observed in melt processed samples [14], namely, parallel platelets of 123 aligned in a preferential direction with 211 particles distributed in them. Some Sm-211 free regions are also seen in the micrographs, and their sizes decrease with increasing 211 concentration. Kim et al. [15] have explained the origin of 211-free regions on the basis of oxygen gas evolution during melt processing. Due to a change in the valency of copper during melting, oxygen gas is evolved, and a pore is formed. Since the mobility of liquid phases is more, the pore is likely to be filled by it than by the solid 211 particles. After the peritectic reaction is over, a 211-free region is formed at the pore site. The morphology of the trapped 211 inclusions in the samples change with increasing 211 concentration. In the stoichiometric and the 10 mol % Sm-211 containing sample, the 211 particles have an acicular morphology with a high aspect ratio of ~ 4 to 5. In the 20, 30 and 40 mol % Sm-211 containing samples, the Sm-211s have nearly spherical morphology. In Fig. 3.5, a histogram of Sm-211 particle size distribution in the microstructure is plotted, they show a refinement of 211 particles with increasing 211 concentration, except for the 40 mol % sample in which particles are slightly coarsened.

The microstructural features observed above can be explained on the basis of the increasing number of 211 particles in the pro-peritectic liquid with increasing 211 concentration. To explain the microstructural features, the samples after the melting stage (i.e. from 1115°C) were quenched to room temperature and the nature of 211 particles in the peritectic liquid was observed. Fig. 3.6 shows the
Fig. 3.5: Histogram plots of the $^{211}$ particle size distribution in the micrographs.

(a). 20 mol % Sm-$^{211}$, (b). 30 mol% Sm-$^{211}$, (c). 40 mol % Sm-$^{211}$. 
Fig. 3.6: SEM micrographs of samples quenched from 1115°C to room temperature. (a) Sm-123, Sm-123 with (b) 10 mol % Sm-211, (c) 20 mol % Sm-211.
SEM micrographs of the samples of Sm-123 and Sm-123 containing 10 & 20 mol % Sm-211. It can be seen that the first two samples with lower Sm-211 content have large acicular 211 particles after the melting stage. In the 20 mol % sample, the size of the 211 particles has decreased and they are blocky. The features seen in Fig. 3.6 seem to explain the microstructure of the melt processed samples. The 211 particle size seen in the first two melt processed samples have acicular shape with large aspect ratios because of the shape of 211 particles at the pro-peritectic stage. After the peritectic reaction, the edges of the 211 particles get rounded off due to its partial dissolution of it during the growth process, but the morphology to a large extent remains same. In the samples with higher concentration of 211 (i.e. 20 to 40 mol %) a large number of 211 particles occur in the pro-peritectic liquid and they do not have enough space to grow with their natural crystallographic habitat into long rods. In the resulting microstructure the 211 particles appear to be refined. The average particle size of the 211s trapped in the microstructure of the melt textured samples and the nucleated 211s at the peritectic melting stage is calculated using standard metallographic techniques [16], and is plotted in Fig. 3.7. The figure shows that with increase in 211 concentration the particle size of it in the microstructure decreases. This is due to the size of the nucleated 211s at the melting stage, for low concentrations it is large and for higher concentrations it is small. In Y-123 system, it has been demonstrated that the smaller sized 211 particles are surrounded by crystallographic defects [17,18]. The defects play an important role in enhancing the $J_c$ as discussed earlier. It remains to be seen in the Sm-123 system whether the $J_c$ is higher when the microstructure contains a large
concentration of small sized Sm-211 particles. However, there is a sudden change above the 10 mol % sample. All the microstructural features change drastically above this concentration. It will be seen below that the magnetic properties also change drastically above the same concentration.

The 211-free regions observed in the microstructures of the melt processed samples can also be explained from Fig. 3.6. The figure shows large areas near the long 211 particles, devoid of 211s. The inhomogeneity in the packing of 211 particles either occurring at the stage of powder consolidation or at the stage of melting due to the development of large acicular 211 particles prevents a packing to 100% density at the melting stage.

![Graph showing 211 particle sizes for increasing 211 concentrations](image)

Fig. 3.7. The 211 particle sizes for increasing 211 concentrations, after the high temperature melting and after melt processing.
Fig. 3.8: Plot of \textit{211-free} regions observed in the microstructures of samples with increasing \textit{211} concentration.

It has been shown by Sudhakar Reddy \textit{et al.} [19], that almost all the shrinkage during melt processing occurs at the melting stage itself and very little during the subsequent peritectic formation of 123 while cooling. At the peritectic reaction stage below $T_p$, the inhomogeneous regions in the 2-phase mixture, without \textit{211} get converted to 123 with the absence of \textit{211} inclusions. With increasing \textit{211} concentration, the \textbf{acicularity} of the \textit{211} particles decreases. This can be expected to result in a reduction in size of the \textit{211-free} regions with increasing \textit{211} content, as observed in our experiments. The same was observed in Fig. 3.8, which is a plot of \textit{211-free} regions with increasing \textit{211} concentrations.
3.3. dc MAGNETIZATION STUDIES

The dc magnetic hysteresis (M-H) loops of the samples processed in low oxygen atmosphere were recorded at 5 K and 77 K for fields applied parallel to the c-axis. The loops are shown in Fig. 3.9. At 5 K, there are no anomalous feature present in the loops. At 77 K for the stoichiometric & 10 mol % sample, have a 'fishtail' or 'peak-effect' at intermediate fields. The effect is more pronounced in the 10 mol % sample. This peak-effect is absent for the rest of the samples at 77 K. The origin of peak-effect was explained by Murakami et al. [20] and Egi et al. [21], as due to the field-induced pinning from the low-\( T_c \) solid-solution regions. Nakamura et al. [22], have reported to have observed a tweed morphology within the twin regions of, single crystal Nd-123 sample. It was also mentioned that the features resemble the spinodal decomposition of an aged alloy, with a modulated structure distributed on a nanometer scale. These structures were assumed to be the low-\( T_c \) solid-solution regions, which contribute to the field-induced pinning, by turning normal at high fields. It was observed in the Y-123 single crystals, that the microstructure consists of a dilute mixture of ortho-I (\( T_c = 90 \) K) & ortho-II (\( T_c \sim 60 \) to 90 K) structures generated by spinodal decomposition [23]. Because of the different superconducting properties of the structures the oxygen-deficient regions have been proposed as flux pinning centers [23-25].
Fig. 3.9(a) : M-H loops of the samples with increasing Sm-211 concentration at 5 K for field applied parallel to c-axis.
Fig. 3.9(b) : M-H loops of Sm123 and Sm-123 with 10 mol % of Sm-211 at 77 K for field applied parallel to c-axis.
Fig. 3.9(c) : M-H loops of Sm123 with 20 and 30 mol % of Sm-211 at 77 K for field applied parallel to c-axis.
Fig. 3.9(d) : M-H loops of Sm123 with 40 mol % of Sm-211 at 77 K for field applied parallel to c-axis.
3.4. TEM STUDIES

To isolate the origin of the peak-effect in the light rare earth 123s, TEM studies were done on the samples. The 10 mol % sample having a pronounced peak-effect, and the 20 mol % sample having no such effect was selected for the TEM studies. The TEM image (Fig. 3.10(a)) of the 10 mol % sample, at high magnification has micro-twins, stacking faults and dislocations. Fig. 3.10(b) shows the dark field image of the sample which is in contrast to the bright field image of Fig. 3.10(a). This indicates that the features observed are not due any sample artifacts like, thickness, bending of the sample, etc. Though a tweed contrast could not be observed the features within the twins at high magnification, shows a dilute concentration of a second phase. The features present in the TEM images resemble that reported in ref. 22. The exact compositions could not be obtained due to the limitations in the TEM setup. The small area electron diffraction (SAD) pattern within the twin region (Fig. 3.10(c)), did not contain any satellite spots, indicating the absence of modulated structures. The pattern was indexed to the orthorhombic structure of Sm-123, with no spots left unindexed. Fig. 3.10(d) shows the electron diffraction pattern from the second phase present in the sample. As the concentration of the second phase is on a finer scale, faint streaks around the central bright spot are visible in the SAD pattern. The presence of streaks around the central bright spot indicates that the second phase observed in the TEM images is the ortho-II structure. Khachaturyan et al. [23] have shown that the presence of diffraction maxima at
Fig. 3.10. (a) TEM image of the 10 mol% sample at high magnification. TB = Twin Boundary, SF = Stacking Fault, DS = Dislocations.

(b) Dark field image of the sample, the image is a contrast to the Fig. 10 (a).
Fig. 3.10. (c) SAD pattern within the twin region of the sample. The zone axis is [010], and the a- & c- axis lie in the plane of the image.

(d) SAD pattern in a different region of the sample. Faintly visible are streaks around the central bright spot, due to the fine distribution of the ortho-II phase.
generic points is a feature of the ortho-II structure. It has also been mentioned by them, that if the 123 sample is cooled very slowly while being oxygenated, secondary tweed structure will form within the twins by an oxygen ordering, which will contain the ortho-I and ortho-II structures. The presence of ortho-II structure has been reported in Y-123 by many groups [24], and also in oxygen depleted Y-123 sample [25], in which the oxygen depletion was due to irradiation effect. In the present study on the Sm-123 sample, the possibility of irradiation in the TEM was minimized by using an accelerating voltage of 160 kv, which is below the value which would have irradiated the sample [26]. The ortho-II structure is an oxygen deficient 123 with a doubling of the unit cell [24]. This leads to the conclusion that the two structures present are: ortho-I with a \( T_c \) above 90 K and ortho-II structure with a \( T_c \approx 60 \) to 90 K. The ortho-II structure in the sample is distributed on a nanometer scale and can be a source of flux pinning at 77 K in high fields, as it turns normal.

In support of the above it may be mentioned that, Khachaturyan et al. [27] have reported that it is thermodynamically possible for the 123 phase to decompose 'spinodally' around 500°C during oxygenation. As it passes sequentially through a series of stoichiometric compositions of Magneli-type phases, a cell doubled ortho-II structure having oxygen deficiency will result. Thus it is possible that "spinodal decomposition" into ortho-I and ortho-II structure is also taking place in the LRE-123 systems around 500°C during oxygenation. The idea of field-induced pinning from the low-\( T_c \) solid-solution regions may not be valid, the present data did not indicate the presence of such regions. The solid-solution regions will have lower c-
lattice parameter because of the substitution of Ba-atom by rare-earth atom [13]. But in the SAD patterns the values of c-parameters were identical. Also, the solid-solution regions would appear in the SAD pattern as different reflection spots, but no such extra unindexed spots were present.

Similar studies were done on the sample having a starting composition of 20 mol % of Sm-211. The TEM image of this sample shows the presence of twin boundaries (Fig. 3.1 l(a)). Fig. 3.1 l(b) shows the SAD pattern for the sample, and the satellite reflections present in the pattern is indicative of the presence of twins in the sample. The SAD pattern recorded within the twin region is shown in Fig. 3.1 l(c), the pattern was indexed to orthorhombic Sm-123 phase. The high magnification picture (Fig. 3.1 l(d)) within the twin boundaries do not have a second phase as observed in the 10 mol % sample, but has micro-twins within the twin boundaries. The data clearly shows that a spinodal decomposition is not taking place in the sample containing 20 mol % Sm-211.

It is known that [28], for an oxide alloy to decompose spinodally, the three important controlling parameters are composition of the alloy, the annealing temperature and elastically soft directions of the solid. Variations in any one of them may disallow the spinodal decomposition. In the literature, it is reported that stoichiometric melt processed RE-123 samples and single crystals show the peak-effect, because of the spinodal decomposition during oxygenation. Here it is shown that the 10 mol % 211 sample also exhibits this phenomenon. The sample with 20 mol % 211 is not having the peak-effect because of the absence of the ‘spinodal decomposition’. The exact reason for absence of ‘spinodal decomposition’ in this
Fig. 3.11. (a) TEM image of the 20 mol % sample showing the twin boundaries in the sample at low magnification.

(b) SAD pattern of the sample having satellite reflection spots, indicating the presence of twins in the sample.
Fig. 3.11. (c) : SAD pattern of the 20 mol % sample within the twin regions of the sample, indicating clearly the structure of Sm-123.

(d) : High magnification TEM picture of the sample, having no second phase distribution within the twin regions of the sample.
sample is difficult to isolate; because many factors are involved as mentioned above. But the origin of the field-induced pinning contributing to the peak-effect or the fishtail effect in the LRE-123 samples is identified to be from the low-$T_c$ oxygen deficient regions, as observed in the Y-123 samples. The oxygen deficient regions do not contribute to the pinning at low fields and in low temperatures, due to the proximity effect coupling between the ortho-I & ortho-II regions. At high fields and high temperatures, the oxygen deficient regions are driven into their normal state because they possess lower $H_{c2}$ and $T_c$, than the oxygen rich regions. Those normal regions contribute to flux pinning, resulting in an increase in $J_c$ near such fields at 77 K.

3.5. TEMPERATURE VARIATION OF M-H LOOPS

Fig. 3.12 shows the magnetization hysteresis loop from 60 to 86 K for field applied parallel to c-axis for the samples exhibiting peak-effect. They are Sm-123 and Sm-123 containing 10 mol \% Sm-211 labeled in this section as sample No. 1 & 2. Sample No. 2 has a pronounced peak-effect in comparison to No. 1. It is clearly visible that the peak in the M-H loop shifts to low fields with increase in temperature. The peak at 60 K is around 5 Tesla region, and the peak gradually moves to lower fields with increase in temperature. This due to the fact that the low-$T_c$ regions which contribute to pinning become normal at lower fields at higher temperatures.
Fig. 3.12(a) : Magnetization hysteresis loop of Sm-123 sample with 10 mol % of Sm-211 from 60 K to 77 K, for field applied parallel to c-axis.
Fig. 3.12(b) : Magnetization hysteresis loop of Sm-123 sample with 10 mol % of Sm-211 from 80 K to 86 K, for field applied parallel to c-axis.
Fig. 3.12(c) : Magnetization hysteresis loop of Sm-123 sample from 60 K to 77 K, for field applied parallel to c-axis.
Fig. 3.12(d) : Magnetization hysteresis loop of Sm-123 sample from 77 K to 82 K, for field applied parallel to c-axis.
3.6. CURRENT DENSITY & FLUX PINNING

In Fig. 3.13 the $J_c$-$H$ relations are plotted for the samples at different temperatures. The $J_c$ increases for low additions of Sm-211 concentration in the starting material. The 10 mol % sample has a higher $J_c$ than the stoichiometric sample. The $J_c$ drops for the 20 mol % sample and again it increases slightly for the 30 mol % sample.

Fig. 3.13(a): $J_c$-$H$ relations for the samples with increasing Sm-211 content at 5 K.
Fig. 3.13(b) : $J_c-H$ relations for the samples with increasing Sm-211 content at 77 K

The 40 mol % sample has a lower $J_c$ than the 30 mol % sample. The $J_c$ at 77 K for 1 Tesla field is plotted in Fig. 3.14, it shows that the $J_c$ has a major peak at the 10 mol % sample and has a minor peak at the 30 mol % sample. It is reported that the $J_c$ in RE-123 (RE= Y to Gd ) increases with 211 concentration, and reaches a maximum around 30 mol % of 211 [29,30], for further increase in 211 concentration the $J_c$ drops to lower values due to saturation of the microstructural features. This $J_c$ behaviour was explained from the microstructures of the samples, with increase in 211 concentration the 211 particles were reduced to a smaller size and thus enclosed large amounts of defects. These defects played a crucial role in increasing the $J_c$. The present experimental results show that the LRE-123 samples
have a different $J_c$ behaviour for increasing $\text{Sm-211}$ concentration. For low additive.

$\text{Sm-211}$ concentration the $J_c$ increases due to the presence of field induced pinning from the low-$T_c$ ortho-II regions which seem to play a major part in increasing the $J_c$. The $J_c$ decreases for the 20 mol % sample because of the absence of field induced pinning. Also the sample is free of crystallographic defects in the matrix, as observed in the TEM images. The second minor peak in $J_c$ is at 30 mol %, which can be due to the optimal refinement of $\text{Sm-211}$ particles in the microstructure. The small sized $\text{Sm-211}$ particles may be surrounded by crystallographic defects like stacking faults etc, which may had increased the $J_c$, like in the RE-123 case. However, the $J_c$ increase for 30 mol % as observed in the Y-123 samples is not present here. The $J_c$ at zero fields is higher for the samples with high concentration of $\text{Sm-211}$, but it reduces to low values with increase in field. The $J_c$-$H$ behaviour with increase in $\text{Sm-211}$ concentration for Nd-123 melt textured samples is reported to be similar [31], as observed in our experiments. This leads to the conclusion that the $J_c$-$H$ behaviour with increase in $\text{Sm-211}$ concentration for LRE-123 melt textured samples is different in comparison to other RE-123 samples.

The pinning force is calculated using the relation $F_p = J_cB/10$, and is plotted against the field in Fig. 3.15. The figure shows that the pinning force is higher for the stoichiometric and the sample containing 10 mol % Sm-$\text{Sm-211}$ sample. The samples containing 20, 30 & 40 mol % of Sm-$\text{Sm-211}$, have very low pinning force. The higher pinning force in the samples with low $\text{Sm-211}$ concentration is because of the reason given above.
Fig. 3.14: $J_c$ at 1 Tesla field at 77 K for the samples vs the mol percentage of added $^{211}$ concentration.

The experimental results indicate that addition of an optimum $^{211}$ concentration (around 10 mol %) in the LRE-123 enhances $J_c$, and for higher concentration it reduces to low values. This result is different to the reported values of concentration required to enhance $J_c$ in RE-123 melt textured samples, where an addition of 30 mol % enhanced the $J_c$.

The pinning force for the two samples (Sm-123 and Sm-123 with 10 mol % $^{211}$) showing the peak-effect is plotted against the field from temperatures of 4 K to 80 K in Fig. 3.16. The figure shows that the Sm-123 sample (sample 1) has a higher pinning force than sample with 10 mol % $^{211}$ (sample 2) in the temperatures below 60 K. Above 60 K sample 2 has a higher pinning force in
comparison to sample 1. The data confirms that the oxygen deficient regions are responsible for the anomalous pinning in the M-H loop, as it enhances the $J_c$ and pinning force above 60 K and below $T_c$ in high fields.

Fig. 3.15: Variation of the flux pinning force with field for the samples with increasing Sm-211 content at 77 K.
Fig. 16(a) : Flux pinning force of sample 1 (Sm-123) from the temperatures 4K. to 60 K.
Fig. 16(b): Flux pinning force of sample 1 (Sm-123) from the temperatures 70K to 80K.
Fig. 16(c) : Flux pinning force of sample 2 (Sm-123 with 10 mol % Sm-211) from the temperatures 4K to 60 K.
Fig. 16(d) : Flux pinning force of sample 2 (Sm-123 with 10 mol % Sm-211) from the temperatures 60 K to 80 K.
3.7. IRREVERSIBILITY LINE

The irreversibility line (IR line) divides the region below which the properties are reversible that which they are irreversible. The slope of the IR line is an indication of the pinning strength. For higher pinning strength, the slope will be much steeper [32]. Fig. 3.17 shows the IR line for the two samples. Sample 2 has a higher slope (-0.69) than sample 1 (-0.46) in the temperature range of 70-90 K. This indicates that the pinning strength has enhanced in that temperature range, due to the presence of the field induced pinning as discussed above. This result is in agreement with the above calculated pinning force, which showed a higher pinning strength for sample 2 above 70 K. The IR line has a point of inflection above which the slope changes to a much higher value. The features of it will be discussed in the next section.

3.8. NORMALISED CREEP RATE

The magnetic relaxation is important as it sets limits to the stability of superconducting devices. The current density of a superconducting sample is dominated by pinning in low fields and relaxation in the high fields. The normalised relaxation rate eliminates the fundamental parameters, and is directly derived from measurements of dM/d(lnT) ([33] and the references in that review). The variation of normalised relaxation rate with field at 77 K is plotted in Fig. 3.18 for the two samples. The relaxation rate of sample 1 increases with field, and for sample 2 the relaxation rate decreases to a very low value in the 1-2 Tesla region, and thereafter increases with field. The value of the field at which the minimum in
the relaxation rate has reached (1.2 T), is equal to the field value at which the slope of the IR line changes to a higher value. In Fig. 3.19, the \( J_c \), flux pinning force and relaxation rate, at 77 K for sample 2 are plotted together. It shows that the field at which the peak in \( J_c \) is obtained is not coincident with the value at which there is a drop in the relaxation. The field value at which the peak in \( J_c \) is obtained is approximately equal to double the value at which a minimum in relaxation rate has reached. The flux pinning force has a peak around 4 Tesla. This indicates that a saturation of pinning is taking place at that field value, above that field value the relaxation mechanism dominates and below it the pinning mechanism plays an important role. It also indicates that the peak in the hysteresis loop is not due to a decrease in the relaxation rate. The alternative is to consider the field induced pinning from oxygen deficient regions.

In Fig. 3.20, the \( J_c \), pinning force and relaxation rate at 77 K for sample 1 are plotted together. It can be seen that as the relaxation rate increases the \( J_c \) decreases. Also, wherever there is a slope change in the relaxation rate, correspondingly there is a drop in the \( J_c \) at same field value. Initially the relaxation rate drops because the flux has to enter the specimen and has to get redistributed. During this the relaxation drops to a low value. The relaxation rate then increases linearly with field. At a field of 0.9 Tesla there is slope change, this corresponds to the field at which there is a slope change in the IR line. The slope of the relaxation rate again changes at a field value of 2.3 Tesla, this corresponds to the peak in the flux pinning force. A saturation of pinning takes place at this field value and above it the relaxation completely dominates and it increases linearly. Fig. 3.19 and 3.20 clearly indicate that in low fields the behaviour of \( J_c \) is dependent on pinning and in
high fields the relaxation mechanism dominates it. The low relaxation rate at 77 K for sample 2 in comparison to sample 1, is due to the predominance of field induced pinning from the low $T_c$ oxygen deficient regions. The drop in relaxation rate and a steeper IR line help to operate the superconducting devices at higher temperatures and fields. This is because sustainable current density can be maintained even under those conditions. But it is difficult to tailor make a sample with the required amount of defects. Irradiation of the sample might help to further lower the relaxation rate as it produces columnar defects of a smaller dimension [34].

Fig. 3.17: Irreversibility line for samples 1 and 2 for field applied parallel to c-axis.
Fig. 3.18: Variation of the normalised relaxation rate with field at 77 K for the samples 1 & 2.

Fig. 3.19: Plot of $J_c$, pinning force and relaxation rate with field for sample 2 at 77 K.
Fig. 3.20: Plot of $J_c$, pinning force and relaxation rate with field for sample 1 at 77 K.
REFERENCES


