Magnetization and magneto-resistance in Y (Ba$_{1-x}$Sr$_x$)$_2$Cu$_3$O$_{7-\delta}$ ($x = 0.00–0.50$) superconductors

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Abstract

Here we present the magnetic properties and upper critical field ($B_{C2}$) of polycrystalline Y (Ba$_{1-x}$Sr$_x$)$_2$Cu$_3$O$_{7-\delta}$ superconductors, which are determined through detailed ac/dc susceptibility and resistivity under magnetic field study. All the samples are synthesized through the solid state reaction route. Reduction in the Meissner fraction (the ratio of field cooled to zero field cooled magnetization) is observed with increasing Sr content, suggesting the occurrence of flux pinning in the doped samples. The ac susceptibility and resistivity measurements reveal improved grain coupling in Sr substituted samples. Consequently the inter-grain critical current density ($J_c$), upward curvature near $T_c$ in the temperature dependence of the upper critical field ($B_{C2}(T)$), and $B_{C2}$ are enhanced. Both $J_c$ and $B_{C2}$ increase in lower Sr substitution (up to $x = 0.10$) samples followed by decrease for higher doping due to degradation in the effective pinning and grain coupling.

(Some figures may appear in colour only in the online journal)

1. Introduction

The application of external mechanical pressure on layered cuprate superconductors is an important tool in engineering an increase in their transition temperature ($T_c$). The increase of $T_c$ depends upon the chemical composition of the parent phase. On the other hand, another strategy, which leads to optimization of the physical properties of a given superconducting system, is cation/anion substitution. Therefore reproduction of the structural changes and further increase in the physical properties which are induced by mechanical pressure, can also be achieved by appropriate chemical substitutions. An example is the partial substitution of Sr cations ($r_1 = 1.31$ Å) at the Ba ($r_1 = 1.47$ Å) sites in YBa$_2$Cu$_3$O$_{7-\delta}$ (Y-123) cuprate superconductor. But substitution of Ba by Sr in Y-123 does not increase the number of carriers in the CuO$_2$ planes as both are divalent. However, it causes a decrease in $T_c$ and results in an unstable 123 phase. In almost all cuprates, $T_c$ decreases when Ba cations are replaced by Sr, except for (La$_{1-x}$Ba$_x$)$_2$CuO$_4$ [1].

Several explanations have been proposed to account for the occurrence of this negative coefficient ($dT_c/dx$) in Y (Ba$_{1-x}$Sr$_x$)$_2$Cu$_3$O$_{7-\delta}$. Licci et al [2, 3] proposed a mechanism in terms of bond valence sum (BVS) for Ba atoms, which decreases with Sr substitution due to the reduction in ionic size. This prevents charge transfer from the Cu(1) to the Cu(2) site and consequently results in decreased $T_c$. According to Ono et al [4] the O content decreases slightly in Sr doped samples due to structural disturbances. Along with decrease in the O content, the reduction in cell volume causes a decrease in $T_c$ [2–6]. Veal et al [5] accounted for this $T_c$ reduction as due to local structure distortions occurring due to Sr doping. On the other hand Zhang et al [7] concluded using x-ray absorption fine structure (XAFS) that local structure...
distortions were not responsible for the reduction in $T_c$ and the entity of the CuO$_2$–Y–CuO$_2$ sandwich structure in Sr doped and un-doped samples was more important.

Although Sr substitution shows a negative effect on $T_c$, it is interesting to investigate how it affects other physical parameters like $J_c$ and $B_{C2}$, which are useful in practical applications of these materials [8, 9]. In this investigation we have explored major limiting factors on these parameters such as inter-grain weak links and poor flux pinning. We have determined the inter-grain critical current density $J_c$ and the upper critical field through resistivity under magnetic field measurements. It is found that moderate Sr substitution causes flux pinning with enhancement of grain coupling, which could enhance both $J_c$ and $B_{C2}$.

2. Experimental details

Samples of Y (Ba$_{1-x}$Sr$_x$)$_2$Cu$_3$O$_{7-δ}$ (with nominal compositions of $x = 0.00, 0.025, 0.05, 0.075, 0.10, 0.25$ and $0.50$) were synthesized through the conventional solid state reaction route. High purity (99.99%) powders of Y$_2$O$_3$, SrCO$_3$, BaCO$_3$ and CuO with a stoichiometric ratio were mixed in an agate mortar. After initial grinding, calcination was carried out in alumina crucibles at 860 °C in air for 36 h. Subsequent calcinations were carried out at 870, 890 and 910 °C temperatures for the same duration with intermediate grinding. In each calcination cycle cooling was carried out slowly and the samples were re-ground well before the next cycle. After final calcination, the samples were pressed into rectangular pellet form and sintered at 925 °C for 48 h in air. Finally the samples were annealed with flowing oxygen at 750 °C for 12 h, 600 °C for 24 h and 450 °C for 24 h and subsequently cooled in 6 h. The phase formation was determined through x-ray powder diffraction, using a Rigaku x-ray diffractometer (Cu Kα). Rietveld analysis of all samples was performed using the Fullprof program. The ac/dc susceptibility ($\chi$–$T$), magnetization ($M$–$T$) and resistivity measurements with and without field ($RTH$) were made by a physical properties measurement system (Quantum Design—USA PPMS-14 T). The scanning electron microscopy (SEM) images of the samples were taken on a Zeiss EVO MA-10 scanning electron microscope.

3. Results and discussion

Table 1. Rietveld refined parameters and orthorhombic distortion of Y (Ba$_{1-x}$Sr$_x$)$_2$Cu$_3$O$_{7-δ}$ oxygen annealed samples.

| $x$   | $a$ (Å) | $b$ (Å) | $c$ (Å) | $R_p$ | $R_w$ | $\chi^2$ | $S$ (with) |
|-------|---------|---------|---------|-------|-------|----------|------------|
| 0.00  | 3.828(6)| 3.886(5)| 11.688(7)| 5.34  | 7.02  | 3.68     | 0.0150     |
| 0.025 | 3.830(5)| 3.893(2)| 11.698(7)| 4.90  | 6.20  | 2.91     | 0.0161     |
| 0.05  | 3.828(1)| 3.887(1)| 11.683(5)| 4.96  | 6.25  | 2.50     | 0.0153     |
| 0.075 | 3.823(2)| 3.886(3)| 11.673(5)| 4.53  | 5.68  | 2.48     | 0.0163     |
| 0.10  | 3.817(2)| 3.879(4)| 11.654(8)| 5.02  | 6.31  | 3.07     | 0.0164     |
| 0.25  | 3.811(3)| 3.876(1)| 11.631(7)| 5.10  | 7.30  | 5.23     | 0.0171     |
| 0.50  | 3.800(9)| 3.856(6)| 11.592(5)| 6.20  | 9.41  | 10.7     | 0.0158     |

Figure 1. Rietveld fitted powder XRD patterns of Y (Ba$_{1-x}$Sr$_x$)$_2$Cu$_3$O$_{7-δ}$. $x = 0.00, 0.025, 0.05, 0.075, 0.10, 0.25$ and $0.50$ oxygen annealed samples.
Figure 2. SEM images of Y_{(Ba_{1-x}Sr_x)_{2}}Cu_{3}O_{7-δ} for (a) x = 0.00, (b) x = 0.05, (c) x = 0.10 and (d) x = 0.25 samples at 5000 magnification.

also given in table 1. According to the calculated values, the orthorhombicity is slightly increased with x and is maximum for x = 0.25. For x = 0.50 it again decreases in agreement with earlier studies [2–6]. However, small impurity phases arises in the x = 0.50 composition as the increasing Sr content results in an unstable 123 phase. SEM images of freshly fractured samples with x = 0.00, 0.05, 0.10 and 0.25 are given in figures 2(a)–(d). It can be observed that the doped samples consist of slightly smaller grains than the pristine sample in agreement with [6]. Also the doped samples show better surface texture with lower levels of porosity than the pristine sample. These observations are consistent with improvement of the grain coupling with Sr doping.

Variation of the dimensionless dc volume susceptibility, in both field cooled (FC) and zero field cooled (ZFC) conditions is shown in figure 3. A decrease in Tc with increasing Sr content can be seen. There is a remarkable increase in separation between the FC and ZFC signals along with decreasing FC signal. This variation is monotonic up to x = 0.10. The increase in separation of the FC and ZFC signals or in other words reduction in the Meissner fraction (ratio of field cooled to zero field cooled magnetization) is a clear indication of flux pinning [10]. This may enhance the critical current and critical fields. For compositions with x > 0.10 the FC and ZFC signal separation is reducing. The saturated magnetization of the ZFC signal is a measure of the diamagnetic shielding current, which initially increases with Sr content and then decreases for higher doping levels.

The ac magnetization of all these samples measured at different ac field amplitudes with zero bias dc field is given in figures 4(a)–(f). The driven magnetic field is parallel to the long axis of the measured samples. It is well known that the real part of the susceptibility consists of two transitions, which correspond to the flux removal from intra-grain and inter-grain regimes. In accord, the imaginary part contains two peaks, which represent energy dissipation and ac losses due to the flux motion in the intra-grain and inter-grain regions [11–16]. This suggests that the higher temperature peak in the imaginary part, whose amplitude is a measure of grain size [14–16], arises due to the grains, and the lower temperature peak is due to the grain boundaries. Accordingly in figure 4(a) the pristine sample shows dual signal structure in both the imaginary and real part measurements. But none of the doped samples show a clear two peak structure in the imaginary part though it is observed in the real
Figure 4. The real part and the imaginary part of the ac magnetization of $Y(Ba_{1-x}Sr_x)_{2}Cu_3O_{7-\delta}$ for (a) $x = 0.00$, (b) $x = 0.025$, (c) $x = 0.05$, (d) $x = 0.10$, (e) $x = 0.25$ and (f) $x = 0.50$ samples, taken at different ac driven field amplitudes with 333 Hz constant frequency.

part. This might be due to higher inter-grain couplings of the doped samples, which could suppress the intra-grain component. Also in the Sr doped samples, smaller grains are observed in the SEM images (figures 2(a)–(d)), due to which the intra-grain component becomes suppressed [16]. In the pristine sample one can notice that the position (temperature) of the high temperature peak is almost insensitive to the amplitude of the driven field (inset to figure 4(a)), while the position of the low temperature peak is sensitive to the field amplitude. A broadening in the low temperature peak is also accompanied by this [16]. For lower fields, it is difficult to distinguish the intra-grain and inter-grain
regions due to persisting strong inter-grain couplings. But at moderate fields separate inter-grain and intra-grain regimes and well distinguished inter-grain and intra-grain signals are observed [11–16]. For this reason the inter-grain peak in figure 4(a) lies very close to the intra-grain peak at lower fields but it shifts to low temperatures at higher fields.

In the case of Sr doped samples, the shift of the inter-grain peak from the intra-grain peak decreases with Sr substitution from $x = 0.00–0.10$ (see figures 4(b)–(d)). For $x = 0.25$ and 0.50, the shift of the inter-grain peak from the intra-grain peak is further increased (see figures 4(e) and (f)). This clearly shows that the grain coupling of doped Y-123 is improved with Sr content up to $x = 0.10$, and is decreased for the $x = 0.25$ and 0.50 samples. The same is also inferred from the SEM images. For more clarity with regard to the behavior of the inter-grain peak, the ac susceptibility under different driven fields of all these samples is given in figure 5. It is more understandable here that the curves are not only shifted to higher temperature but also the transition width is reduced for the moderately doped samples, up to $x = 0.10$. Clearly the fields (ac amplitudes), which are strong enough to separate inter- and intra-grain interactions in the pure sample, are not strong enough to separate these interactions in moderately ($x \leq 0.10$) doped samples. This indicates that strong inter-grain couplings are induced in the system with the substitution of Sr. Somehow the coupling peak again tends to shift to lower temperatures for the higher doped ($x > 0.10$) samples, indicating decrease of the inter-grain coupling.

Figure 6 shows the real and imaginary components of the ac susceptibility measured at 17 Oe and with a 333 Hz ac driven field. This figure gives an explicit explanation of the relative variation of strength of the inter-grain couplings of doped and un-doped samples. In the real part, the pristine sample shows well separated two step transitions with positive slope at the onset part. In contrast to this, with Sr substitution the second transition shifts towards the higher temperatures and is overlapped with the first transition. This results in a sharp single transition with negative slope at the onset part, indicating a strong coupling component [11–16]. However, with further increase of dopant level ($x > 0.10$) the two step transition again appears, indicating weakening of the grain coupling. In accord, the two peaks in the imaginary part follow the same variation. These observations collectively show that moderately Sr doped samples possess enhanced grain coupling, which may lead to increase in the critical
Figure 7. Variation of $J_c$ with $T_P$ of Y ($\text{Ba}_{1-x}\text{Sr}_x$)$_2\text{Cu}_3\text{O}_{7-\delta}$, $x = 0.00, 0.025, 0.05, 0.10, 0.25$ and $0.50$ samples. The solid lines are just guides to the eye.

current and critical fields. Increase of $J_c$ up to moderate Sr concentrations was also observed in earlier reports [8]. We calculated the temperature dependence of the inter-grain critical current density $J_c(T)$ using the relation $J_c(T_P) = H_a/a$ employing Bean’s model [17]. Here $2a \times 2b$, where $a < b$, is the cross section of the sample, $H_a$ is the amplitude of the applied ac field and $J_c(T_P)$ is the inter-granular critical current density at $T_P$, the temperature of the inter-grain peak. The calculated values are plotted with temperature ($T_P$) in figure 7. $J_c$ is estimated only in the neighborhood of $T_c$, where the shift of the inter-grain peak extends towards lower temperatures within the limit of the driven field. It is seen that the inter-grain current is improved for moderately Sr doped samples and excessive doping cause degradation of the critical current. Both the dc susceptibility (figure 3) and the detailed ac magnetization (figures 4–6) results clearly indicate that the inter-grain coupling of pristine Y-123 is improved for moderately ($x$ up to $0.10$) Ba site Sr doped samples.

Figures 8(a)–(e) depict the normalized resistivity curves under magnetic field ($\rho_{\text{nor}} TH$) up to 13 T dc field, applied perpendicular to the current flow for the Y ($\text{Ba}_{1-x}\text{Sr}_x$)$_2\text{Cu}_3\text{O}_{7-\delta}$ ($x = 0.00, 0.05, 0.10, 0.25$ and $0.50$) samples. In zero field, the $T_c$ ($\rho = 0$) of the $x = 0.00$ sample is around 90 K (figure 5(a)) and it decreases monotonically to around 80 K for the $x = 0.50$ (figure 5(e)) sample. The decrease in $T_c$ ($\rho = 0$) is in agreement with the magnetization results (figures 3 and 4). The $RTH$ curves show (figures 8(a)–(e)) basically two transitions, which is in agreement with our ac magnetization results and earlier reports [18–21]. It can be seen that the effect of magnetic field is weaker at the onset part near the normal state in comparison to the tail part. Also, the offset of $T_c$ ($\rho = 0$) is moved to lower temperatures with increasing field. This occurs near the onset part, where superconductivity persists only inside individual grains and the superconducting fraction is quite small. On the other hand at the tail part, superconductivity persists not only in the grains but also in the grain boundaries, leading to a higher superconducting fraction [22]. A long range superconducting state with zero resistance is achieved by means of a percolation like process that overcomes the weak links between grains [23]. The broadening of the tail part occurs due to the anisotropic nature and disturbances in the percolation path between grains (due to poor grain alignment) caused by applied field [24, 25]. It is also noticeable that the rapid shift of the tail part towards lower temperatures occurs at low fields (below 5 T), afterward
a smaller and almost uniform shift occurs at higher field. This can be explained as being because in lower applied fields with increasing field, poorly oriented grains become normal. But at higher fields, only favorably oriented grains contribute to the superconductivity and thus there is no further broadening. Also in higher magnetic fields, the field tends to penetrate individual grains and this causes broadening of the onset part. As far as the impact of Ba site Sr substitution is concerned, it can be noticed that in Sr doped samples there is a monotonic decrease in the shift of the tail part towards lower temperatures. This may be attributed to enhanced grain coupling in Sr doped samples in consistency with ac/dc susceptibility data. However, for higher doping levels (x = 0.25 and 0.50), the increase in the shift of the tail part may be due to slight weakening of the grain coupling in accord with the ac magnetization data.

Plots of the temperature derivative of resistivity are shown in figures 9(a)–(e). It is well known that the temperature derivative of resistivity gives narrow intense maxima centered at \(T_c\) and a broad peak at low temperatures representing the intra-grain and inter-grain regimes [18–21]. In all plots (figures 9(a)–(e)) a single peak structure appears in zero field confirming that the grains maintain a good percolation path between themselves. On the other hand two peaks are seen under applied field, corresponding to both regimes, separated from each other at different rates with increasing field. The peak corresponding to the granular network shifts towards lower temperatures more rapidly than the bulk peak. Simultaneously rapid broadening of the coupling peak can be seen. The bulk peak is also diminished with increasing field as flux penetrates into the individual grains. It can also be seen that the separation of the coupling peak from the bulk peak has decreased in Sr doped samples. We calculated the temperature difference (\(\Delta T_{P2}\)) between the positions of the coupling peak at 13 and at 0.1 T and plotted its variation with x (see figure 10). This shows that the gap between the two peaks reduces with increase of x up to x \(\leq\) 0.10 and then slightly increases for x > 0.10. By taking all these data into the account (ac susceptibility and resistivity data) it is reasonable to conclude that the moderate substitution of Sr at the Ba site in the Y-123 system causes enhancement of the grain coupling.

The estimation of the temperature dependence of the resistive upper critical field (\(B_{C2}(T)\)) using midpoint data (where the resistivity is half of its normal state value) is shown in the inset of figure 10. It is seen that all the samples show concave curvature (upward curvature) near \(T_c\) followed by a linear region which is in agreement with earlier studies [24–26]. It is also noticeable that this upturn is increased in Sr doped samples compared with the pristine sample. Werthamer, Helfand, and Hohenberg (WHH) theory gives a solution for the linearized Gor’kov equations for \(H_{C2}\) for bulk weakly coupled type II superconductors, including the effects of Pauli spin paramagnetism and spin orbit scattering [27]. Here we use a simplified WHH equation to estimate \(B_{C2}(T)\) without spin paramagnetism and spin orbit scattering.\[B_{C2}(T) = \frac{\alpha}{T} \left(1 - \frac{T}{T_c}\right)^2 \left(1 - \frac{T}{T_c}\right)^{-1}\]

\[\alpha \approx \frac{\pi^2 k_B^2 T_c^2}{\mu_0 H_{C2}^2}\]

where \(\alpha\) is a constant and \(\mu_0\) is the permeability of free space. The inset shows a zoomed picture of the coupling peak.
interaction given by
\[
\ln \frac{1}{t} = \psi \left( \frac{1}{2} + \frac{\tilde{h}}{2T} \right) - \psi \left( \frac{1}{2} \right),
\]
where \( t = T/T_c \) is the digamma function and \( \tilde{h} \) is given by
\[
\tilde{h} = \frac{4H_\text{C2}}{\pi^2 T_c (-\alpha H_\text{C2}/dT)_{T=T_c}}.
\]

Using the slope of the linear portion of the experimental data and the corresponding extrapolated \( T_c \) values to the \( \mu_0H_\text{C2} = 0 \) region, the \( \mu_0H_\text{C2}(T) \) is fitted using the simplified WHH model. The fitted data are shown in figure 11. In accordance with the pinning behavior revealed in dc magnetization measurements and variation of inter-grain coupling strength, the estimated \( B_{\text{C2}} \) value increases with Sr substitution followed by a small decrease. The calculated \( B_{\text{C2}(0)} \) of the pristine sample is around 66 T and with increasing Sr concentration the maximum \( B_{\text{C2}(0)} \) is found to be around 140 T for the \( x = 0.10 \) composition. Several studies have been made earlier on single crystals of Y-123 and \( B_{\text{C2}(0)} \) has been calculated [28–31]. Large anisotropy has been observed for \( B_{\text{C2}(0)} \) when field is applied parallel and perpendicular to the CuO planes [28–30]. Various approximations have been used and the calculated \( B_{\text{C2}(0)} \) varies from 56 to 120 T when field is applied perpendicular to the CuO planes [28–30]. Our findings for the studied samples (66 T for pristine Y-123 and 140 T for the \( x = 0.10 \) composition) seem reasonable in the context of bulk polycrystalline samples.

In summary, Sr substitution at the Ba sites results in an increase of the flux pinning property in Y-123. The effective pinning initially increases with Sr content followed by a slight decrease. The grain coupling behavior in terms of its strength follows the same variation with Sr content. The inter-grain critical current density and resistive upper critical field increase with Sr doping due to the effective flux pinning together with the improved grain coupling nature of doped Y-123 samples.

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