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Hall effect in iron-doped $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ superconductors

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Abstract

Hall effect and resistivity studies have been performed on $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$ superconductor samples (x = 0–0.3) from liquid nitrogen to room temperature. The transition temperature, resistivity and carrier concentration of the doped superconductors are observed to decrease systematically with doping concentration. The Hall number of a unit cell is observed to reach nearly unity at higher concentrations of iron. Similarly to the undoped sample, the Hall coefficient R_H of doped samples varied linearly with temperature until a particular doping concentration (x = 0.07) and then showed a lesser temperature dependence. Different possibilities such as skew scattering, the presence of multiple bands and coupling between Cu–O planes are discussed in order to understand the temperature variation of R_H. The Hall mobility of the carriers is understood as being due to electron–phonon and electron–electron scattering, except for the highest concentration of iron (x = 0.3) when an additional scattering mechanism is expected. The results are explained in terms of defects arising from the substitution of copper by iron.

1. Introduction

Since the discovery of high T_c superconductors by Bednorz and Muller [1], several unusual electronic properties have been observed in both single-crystal and polycrystalline copper-based oxides [2–4]. One of these properties, the Hall effect, has proved the existence of correlations between parameters such as the Hall coefficient, the zero-resistance temperature (T_c) and the concentration of oxygen or cations [2, 5]. It was thus shown that the carrier density $n = 1/eR_H$, where R_H is the Hall coefficient, is strongly related to the superconducting transition temperature T_c in $La_{2-x}Sr_xCuO_4$ [6] and $YBa_2Cu_3O_7$ [2, 5]. With increasing n, T_c increases, reaches a maximum and then decreases as n increases further. In addition, $1/eR_H$ decreases linearly with temperature as does the resistivity for $T > T_c$. Recently, a relation between n and T_c was also shown to exist in $Bi_2Sr_2CaCu_2O_y$ (2:2:1:2) and $Bi_2Sr_2Ca_2Cu_3O_y$ (2:2:2:3) systems doped with different single-element substitutions [2, 7, 8]. Resistivity

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measurements on a single crystal of $YBa_2Cu_3O_{7-y}$ showed that the in-plane resistivity (parallel to the Cu–O plane) is nearly linearly temperature dependent from T_c up to room temperature [9]. Carrier–phonon and carrier–carrier scattering mechanisms have been assumed to explain this behaviour broadly. However, the effect of double dopants on resistivity, Hall number and Hall mobility have not been studied.

Lead doping in bismuth oxide and iron doping in $YBa_2Cu_3O_{7-y}$ are independently known to increase and decrease the transition temperatures respectively [10, 11]. Double dopants in bismuth oxide are interesting to verify the relation between T_c and carrier concentration and the mobility dependence on temperature. In this work Hall effect and resistivity studies are performed on samples of $Bi_2Sr_2Ca_2Cu_3O_y$ superconductors doped with a fixed amount of lead and different amounts of iron in order to observe the changes in carrier density due to double dopants (lead and iron), to examine the T_c–n relation and to determine the temperature dependence of mobility. It is observed that both the transition temperature and carrier density decrease with increasing iron concentration and that the transition temperature decreases with decreasing carrier concentration. The temperature dependence of mobility is attributed to processes of electron–phonon and electron–electron scattering. Evidence of an unknown scattering process is also observed affecting the mobility of carriers in highly doped samples.

2. Experimental details

Samples of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$ ($x=0$, 0.04, 0.07, 0.1, 0.2 and 0.3) are prepared by high temperature solid state reaction. Appropriate amounts of Bi_2O_3, PbO, $CaCO_3$, $SrCO_3$, CuO and Fe_2O_3 powders taken in their molar ratios are thoroughly mixed and ground in a mortar. The mixed powder is prefired in air at 820 °C for 48 h followed by an intermediate grinding. Then it is reground and pressed into pellets 15 mm in diameter and 2–3 mm thick by applying a load of 5 tf. The pellets are sintered at 850–750 °C and then slowly cooled to room temperature. A Philips X-ray diffractometer (PW 1700) with Cu Kα ($\lambda = 1.5418$ Å) radiation is used to record the powder diffraction patterns of the samples. No extra peak corresponding to PbO or Fe_2O_3 is detected.

The d.c. Hall coefficient and d.c. resistivity are measured using the five-probe technique. Samples of uniform thickness are cut into a rectangular bar geometry and the electrical contacts are made with high purity silver paste. All measurements are taken from the onset of the transition temperature to room temperature by mounting the sample in a double-walled vacuum cryostat. The d.c. voltage drop and direct current are measured using a Keithley 196 DMM, while the current is maintained at 0.5 mA using a constant-current source. The temperature is measured to an accuracy of 0.5 °C using a flat strip germanium thermometer in zero magnetic field and a copper–constantan thermocouple in the presence of a magnetic field. The Hall coefficient is measured under a magnetic field H of 8 kG and is considered to be within the low field Hall regime where electron scattering plays a dominant role.

3. Results

Figure 1 shows the temperature variation of resistivity for $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$ samples with $x=0$, 0.04, 0.07, 0.1, 0.2 and 0.3. The curves show almost a linear decrease in resistivity down to 120 K, *i.e.* metallic behaviour, and zero resistivity at different temperatures. The samples with $x=0.1$ and 0.2 show a small tail before attaining the zero-resistance temperature. Owing to experimental limitations, the same could not be observed for the sample with $x=0.3$. However, its transition temperature can be estimated by extending the resistivity plot. The transition temperatures of the different samples are given in Table 1. It is also seen from Fig. 1 that

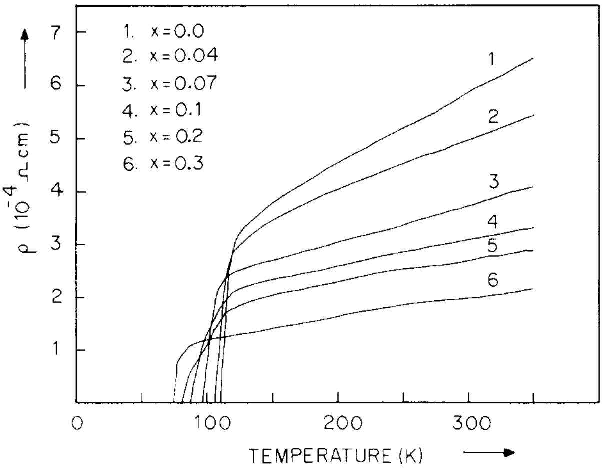

Fig. 1. Temperature dependence of resistivity for $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$ superconductors with $x=0.0$–0.3.

TABLE 1

Transition temperature (T_c), charge carrier concentration at 300 K (p_H) and constants A and B for $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$ superconductors

x	T_c (K)	p_H (10^{21} cm^{-3})	A (10^{-3} K^{-1})	B (10^{-7} K^{-2})
0.00	108	12.0	1.7	69.0
0.04	105	9.2	1.5	32.2
0.07	99	6.1	9.4	8.6
0.10	88	4.4	5.2	6.3
0.20	82	3.3	3.3	4.3
0.30	71	2.3	9.1	5.1

the absolute value of the resistivity and T_c decrease systematically with increasing x, *i.e.* the iron concentration, generally in agreement with earlier work on sintered polycrystals of $YBa_2Cu_3O_y$ doped with iron [11]. The small indication of the presence of a low temperature phase observed in the form of a resistivity tail in the samples with $x=0.1$ and 0.2 was also evidenced in their X-ray diffractograms. However, their transition temperatures are higher than 80 K. In addition, dominant X-ray diffraction peaks corresponding to the low temperature phase are seen in the sample with $x=0.3$. The low T_c (80 K) 2:2:1:2 phase of the sample with $x=0.3$ is characterized by $a=b=5.4$ Å and $c=30.8$ Å, while the high T_c (105 K) 2:2:2:3 phase observed in the undoped sample and the sample with $x=0.04$ has a c parameter of 37.1 Å. Further, in no sample is an extra peak of lead or iron detected. Within the limits of doping concentration the unit cell parameters of the 2:2:1:2 and 2:2:2:3 structures have not shown a significant variation with the substituted lead and iron.

Figure 2 shows the temperature dependence of the Hall number, defined as the charge carrier concentration per unit cell ($p_HV=V/R_He$, measured from the Hall effect, where V is the volume of the unit cell), for $Bi_{1.6}Pb_{0.4}Sr_2$-$Cu_{3-x}Fe_xO_y$ samples with $x=0$, 0.04, 0.07, 0.1, 0.2 and 0.3. The observed positive Hall voltage has evidenced the presence of positive charge carriers in all samples. The samples with $x=0$, 0.04 and 0.07 show a distinctive linear T dependence of p_HV extending up to 300 K, whereas the samples with $x=0.1$, 0.2 and 0.3 show a deviation from linearity. The trend in Fig. 2 also suggests that as x increases, the average slope of $d(p_HV)/dT$ decreases, so that at the highest iron-doping level ($x=0.3$) p_HV is weakly temperature dependent. Over the same range of x the observed values of T_c of different samples decrease quite sharply from 108 to 71 K (Fig. 3). In Fig. 3 the variation in Hall number with composition is also plotted at temperatures of 120, 140, 200 and 300 K. This figure shows that as x increases from zero, the Hall number decreases rapidly and linearly at all temperatures until $x=0.1$ and then saturates to a value close to one hole per unit cell. A similar result was also observed in Y–Ba–Cu–O doped individually with cobalt and nickel [3]. The anomalous temperature variation of the Hall number is nothing but the reflection of the observed anomalous inverse temperature behaviour of R_H. Such an anomaly was also observed in undoped and iron-doped Y–Ba–Cu–O [11–14] and in $Tl_2Ca_2Ba_2Cu_3O_x$ [15], but was inconclusive, because of the complexity. Added to this complexity, in double-doped bismuth oxides the variation in R_H with iron concentration is linear at low temperatures and breaks into two linear parts corresponding to lower and higher concentrations of iron at higher temperatures (Fig. 4).

Since the samples under consideration are all metallic (above T_c), it is quite unlikely that the carrier density decreases with decreasing T. Therefore it should be taken into account that the T dependence of R_H reflects effects other than carrier localization. In metals with magnetic impurities R_H usually shows significant temperature and magnetic field dependence, known as skew scattering [16]. In the present system there is a possibility that Cu^{2+} and

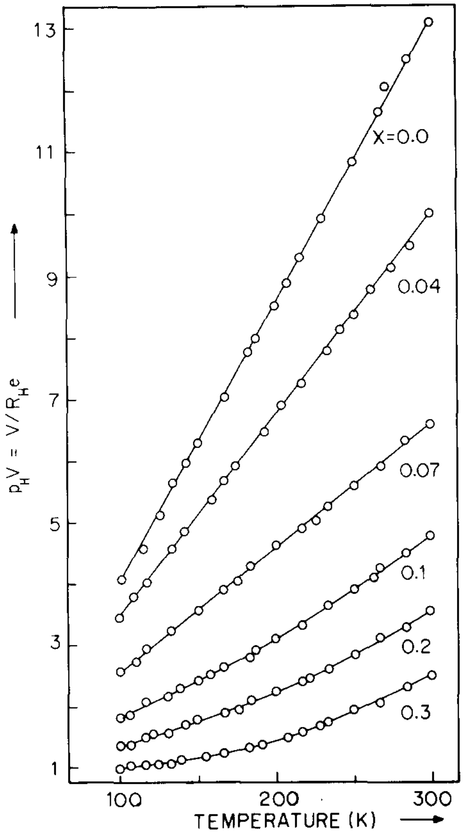

Fig. 2. Temperature variation of Hall number (defined as V/R_He, where V is the unit cell volume) for $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$ with $x = 0.0$–0.3.

a few Fe^{3+} ions may behave as localized magnetic moments and at first sight it is natural to associate the observed temperature dependence of R_H with skew scattering. Thus we measured the magnetic field dependence of the Hall resistance R_{xy} (the Hall voltage divided by the injecting current) of all samples up to 15 kG at different temperatures. The results for the sample with $x = 0.1$ are shown in Fig. 5. The Hall resistance R_{xy} shows a good linear H dependence at temperatures above T_c and no sign of non-linearity is observed. This indicates that there is probably no effect from scattering at least for carriers within the Cu–O and iron-substituted Cu–O planes.

These results do not necessarily mean that there is no local magnetic moment in the $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$ polycrystalline samples. Because skew scattering arises through spin–orbit interaction between conduction carriers and localized moments, the present results might imply that the spin–orbit interaction is very weak in this system.

Figure 6 shows the temperature variation of the Hall mobility μ_H of the positive carriers in $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$ superconductors. From this

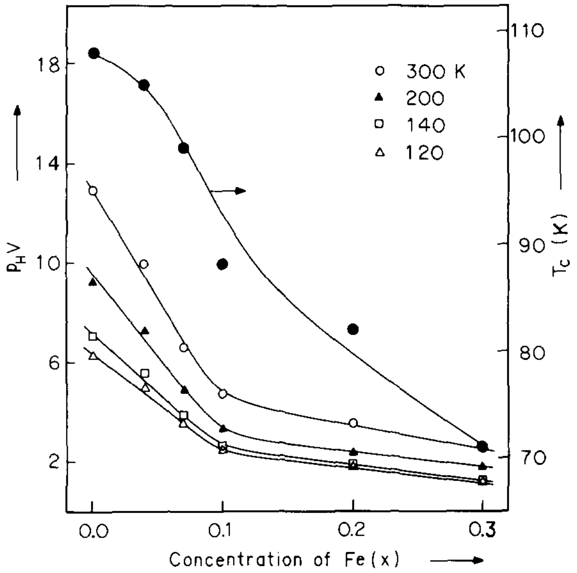

Fig. 3. Top curve: variation in T_c with concentration of iron in $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$. Lower curves: variation in Hall number with x at constant temperature. The data points are interpolations of measurements reported in Fig. 2.

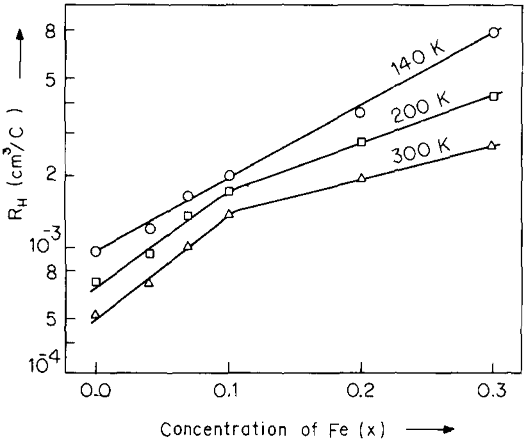

Fig. 4. Variation in Hall coefficient with concentration of iron in $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$. R_H is linear with x at 140 K and shows two linear parts at 200 and 300 K.

figure μ_H is observed to increase with decreasing temperature in all samples, indicating the presence of dominant scattering by phonons. This behaviour is in contrast to $\mu_H(T)$ observed for $BaPb_{1-x}Bi_xO_3$, where doping with bismuth occurs into oxygen octahedral sites and the impurity scattering increases with x at all temperatures. This is due to the apparent decrease in p_H (or increase in R_H) as shown in Fig. 2.

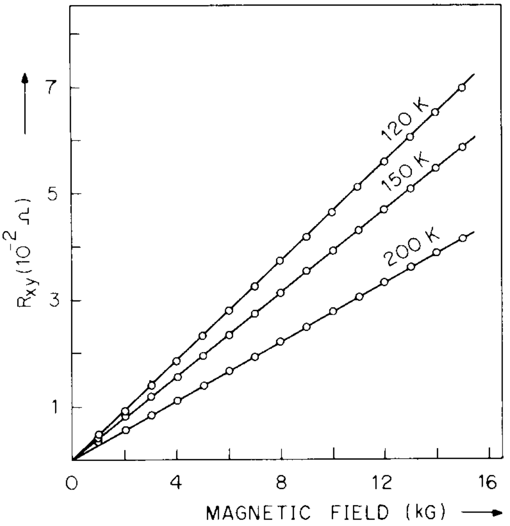

Fig. 5. Magnetic field dependence of Hall resistivity for $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$ with $x = 0.1$ at three different temperatures.

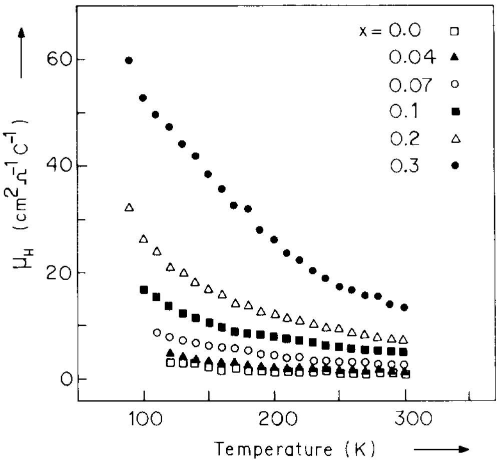

Fig. 6. Temperature variation of mobility for $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$ with (a) $x = 0.0$, (b) $x = 0.04$, (c) $x = 0.07$, (d) $x = 0.1$, (e) $x = 0.2$ and (f) $x = 0.3$.

4. Discussion

The primary interest of the present study is to determine the electronic structure of the Bi–Sr–Ca–Cu–O system (2:2:2:3) doped with the two elements lead and iron. It is observed by Zhang *et al.* [10] that the number of charge carriers decreases quickly at smaller and slowly at larger values of x in the lead-doped Bi–Sr–Ca–Cu–O system. Priftis *et al.* [11] have studied the effect of iron doping in the Y–Ba–Cu–O system and observed that the transition temperature rises as the positive carrier concentration increases, which according to them is due to the rise in stability of bi-polarons. Iron is known in this system as a decreasing agent for hole density. Lead doping in the Bi–Sr–Ca–Cu–O system increases the number of carriers (holes) while iron doping possibly decreases the carrier density by substituting Cu^{2+} by Fe^{3+}. Therefore it is interesting to investigate the transport properties of the Bi–Sr–Ca–Cu–O system doped with both lead and iron. In this work a fixed amount of lead (0.4) and different amounts of iron ($x = 0$–0.3) doping are done in this system and their effects on the transport properties are investigated.

Generally speaking, the Hall effect reflects the dynamics of electronic transport. In the low field regime the Hall effect is explained by collision-dominated transport or, in other words, electron scattering. In our experiments an applied magnetic field $B = 10$ kG corresponds to $B = 10^{-5}$ V s cm^{-2} and $\mu B \ll 1$ is fulfilled. Even at $B = 15$ kG the inequality $\mu B \ll 1$ holds good and therefore the results in Fig. 5 are in the low field regime. As mentioned before, the Bi–Pb–Sr–Ca–Cu–Fe–O system becomes more metallic with increasing x. It is not likely then that the decrease in R_H is caused by carrier localization, at least in the superconducting composition region. Therefore the temperature dependence of R_H seems to reflect the electronic scattering processes. From this point of consideration one should first pay attention to skew scattering by the local magnetic moments which interact with the conduction carriers through spin–orbit coupling. In order to put forth this idea, it is necessary that the susceptibility $\chi(T)$ associated with the local magnetic moment has the same T dependence. However, in the Bi–Sr–Ca–Cu–O system there are few reports about the increase in $\chi(T)$ as T falls. This does not necessarily rule out the existence of local magnetic moments, because the amount of local magnetic moment should be very small owing to the prevailing staggered spin fluctuation in this system. However, the results in Fig. 5 indicate that the interaction between conduction carriers and the local magnetic moment, if any, has a very slight effect on the scattering appearing in the Hall effect.

The temperature dependence of R_H could also be due to the multiple bands in the system. Therefore we shall now look for such possibilities. For a multiband system R_H can generally be written as [17]

$$R_H = \sum R_{Hi}\left(\frac{\sigma_i}{\sigma}\right)^2 \tag{1}$$

where $R_{Hi} = 1/en_i$ and $\sigma_i = n_i\mu_i$ are the Hall coefficient and conductivity of the ith band respectively and n_i and μ_i are the carrier density and mobility of the ith band respectively. For a single band $R_H = 1/en$, which is independent of temperature. For simplicity here we discuss a double-band system:

$$R_H = \frac{n_1 + n_2 b^2}{(n_1 + n_2 b)^2} \tag{2}$$

where $b = \mu_2/\mu_1$. For a metallic system n_1 and n_2 are independent of temperature. The temperature dependence of R_H comes totally from b.

We shall now apply this equation to the Bi–Pb–Sr–Ca–Cu–Fe–O system. By differentiating eqn. (2) with respect to temperature, we get

$$\frac{\mathrm{d}R_H}{\mathrm{d}T}=\frac{2n_1n_2(b-1)}{(n_1+n_2b)^3}\frac{\mathrm{d}b}{\mathrm{d}T} \tag{3}$$

The condition for the right-hand side of the equation to be very small is either (a) $b \approx 1$ or $\mu_1 \approx \mu_2$ in the whole temperature range, which means that the two bands are nearly the same, or (b) $b \gg 1$ or $\mu_1 \ll \mu_2$, *i.e.* band 1 is strongly localized in the whole temperature range. Condition (b) demands a nearly single band whereas condition (a) demands that the two bands have little difference.

From the 2:2:1:2 phase structure of Bi–Sr–Ca–Cu–O we can clearly see that it has two of each of the Cu–O and Bi–O planes in the unit cell. It is natural to ask whether the Bi–O planes make any contribution to the transport and superconducting properties. Because R_H in samples with $x > 0.07$ is weakly dependent on temperature, either condition (a) or (b) needs to be satisfied. If the two bands come from either Cu–O or Bi–O planes, condition (a) can easily be satisfied. However, if one band comes from the Cu–O plane and the other from the Bi–O plane, it is very unlikely that condition (a) will be satisfied because of the difference in valences of bismuth and copper. Therefore condition (b) must be satisfied, *i.e.* the Bi–O plane is strongly localized. For simplicity we will now consider the contribution of the Cu–O plane in the following discussion.

The temperature dependence of R_H could be understood by coupling between Cu–O planes. In the non-coupling case all 2D Cu–O planes form their own energy band and these bands will be degenerate. Coupling will cause a loss of degeneracy. Weak coupling causes weak splitting of the bands; this is what happens in Bi–Sr–Ca–Cu–O. In general we can say that the magnitude of $\Delta R_H/\Delta T$ reflects the strength of the coupling. From Fig. 7 it can be inferred that $\Delta R_H/\Delta T$ for the Bi–Pb – Sr–Ca–Cu–Fe–O system increases up to $x \approx 0.07$ and then remains nearly constant, indicating that the coupling between Cu–O planes is increased by the substitution of iron in place of copper. This is also evidenced by the decrease in transition temperature with doping concentration. Also, iron doping reduces the hole concentration. This can be understood as follows.

There is sufficient evidence from X-ray, neutron, Mössbauer and EXAFS data that iron atoms (for small x) are located at Cu1 sites; then, as Fe^{3+} substitutes for Cu^{2+}, an electron compensates a hole, resulting in a decrease in hole density. However, T_c does not decrease as much as expected in samples doped only with iron. This is because of the presence of lead atoms, which keep the concentration of carriers higher and thus also the transition temperature.

Another possible cause for the significant temperature dependence of R_H may be the coexistence of two types of carriers whose mobility exhibits strong T dependence. In this case R_H is expressed as

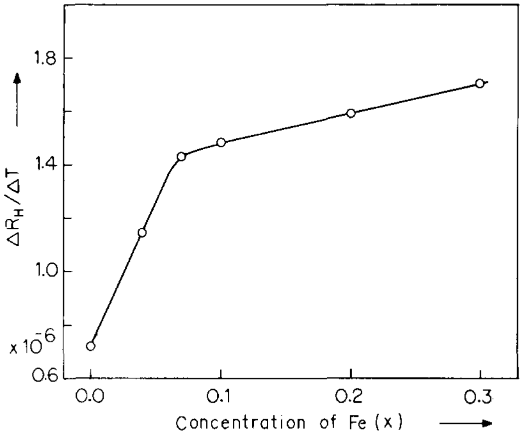

Fig. 7. Variation of $\Delta R_H/\Delta T$ with x. $\Delta R_H/\Delta T$ is seen to increase up to $x \approx 0.07$ and then remain nearly constant.

$$R_H = \frac{\mu_p^2 n_p - \mu_n^2 n_n}{e(n_p\mu_p + n_n\mu_n)^2} \tag{4}$$

As Davidson *et al.* [18] suggested, R_H can be T dependent if n_p, n_n and the mobilities have a T dependence of the form

$$\frac{|\mu_p - \mu_n|}{|\mu_p + \mu_n|} \propto \frac{1}{T} \tag{5}$$

In the case of the Y–Ba–Cu–O system, where the chemical potential is considered to lie close to the midband [19], the above argument holds and characteristics such as $R_H \propto 1/T$ can be expected. In the case of the Bi–Pb–Sr–Ca–Cu–Fe–O system the chemical potential may lie near the midband for $x \leqslant 0.07$ and may deviate thereafter. Therefore the cancellation of n_n and n_p is not adequate for $x > 0.07$, resulting in a moderate temperature dependence. Lu *et al.* [4], while studying the single-crystal Bi–Sr–Ca–Cu–O system, concluded that (i) the Bi–O layer makes little contribution to the conduction and (ii) the temperature dependence of R_H is the result of coupling between Cu–O planes. Hence in the present system the coupling between Cu–O planes may increase with iron doping for $x > 0.07$, resulting in a moderate temperature dependence of R_H. This is also evidenced by the decrease in T_c with increasing iron doping (Table 1).

The unique solution satisfying both the experimental anomalies $R_H \propto 1/T$ and $\rho \propto T$ is that $n \propto T$ and $1/\tau = AT + BT^2$, with A and B related to the constant term ρ and the slope $d\rho/dT$ respectively. Such an expression for $1/\tau$ can be obtained by assuming that $1/\tau = 1/\tau_{e-ph} + 1/\tau_{e-e}$, where the electron–phonon scattering rate $1/\tau_{e-ph}$ has been shown [20, 21] to be nearly linearly T dependent from T_c up to room temperature and the carrier–carrier scattering rate $1/\tau_{e-e}$ is well known to be proportional to T^2 at low temperatures [22]. Thus the relation $1/\tau = AT + BT^2$ seems to be a reasonable approximation,

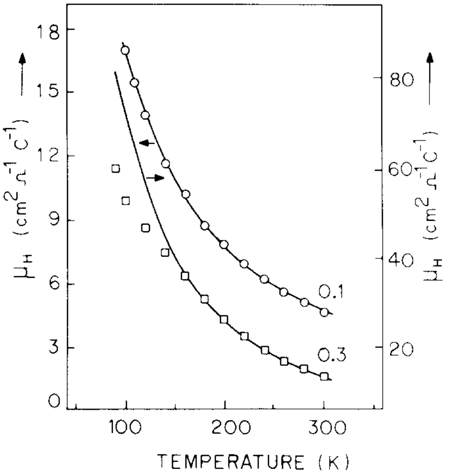

Fig. 8. Temperature variation of Hall mobility of positive carriers for $x = 0.1$ and 0.3. Symbols indicate experimental observations and the continuous line is a fit to the equation $1/\tau = AT + BT^2$. Deviation between experiment and theoretical fit for sample with $x = 0.3$ is seen at low temperatures.

although the coefficient B is expected to be small [22]. A verification of this relation is done by fitting the experimental mobility data with the equation $1/\tau = AT + BT^2$ and the constants A and B are determined. A good fit between the experimental data and the equation is obtained, except for the data corresponding to the sample with $x = 0.3$. A typical fit corresponding to the sample with $x = 0.1$ is shown in Fig. 8. The values of A and B thus obtained for different samples are given in Table 1.

It is seen from Table 1 that the value of B decreases with increasing x until $x = 0.2$ and then increases. Also, for $x = 0.3$ the experimental values of μ_H on the low temperature side are seen to be smaller than the best possible theoretical values based on e–ph and e–e scattering (Fig. 8). This clearly implies that there is an additional scattering mechanism which occurs most probably due to the defects that arise from the iron-substituted Cu–O diamond planes after a doping level of $x = 0.2$. Possibly the differences in size and valence of the iron and copper atoms help the formation of defects. However, this needs further investigation on single crystals.

5. Conclusions

(1) The resistivity and T_c of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}O_y$ superconductors decrease systematically, indicating that the 2:2:2:3 phase gradually changes to 2:2:1:2 with increasing iron concentration.

(2) The Hall number of a unit cell approaches unity at higher concentrations of iron in $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_{3-x}Fe_xO_y$, but the variation in Hall coefficient with iron concentration is linear at lower temperatures and doubly linear at

higher temperatures. This may be due to the ionization of defects related to impurities at higher temperatures. Similarly to the undoped sample, the Hall coefficient of doped samples decreases linearly with increasing temperature until $x = 0.07$ and then becomes less temperature dependent.

(3) Skew scattering is negligibly small in lead- and iron-doped Bi–Sr–Ca–Cu–O superconductors.

(4) Since the Bi–O plane is strongly localized, the possibility of multiple bands (due to Cu–O and Bi–O planes) causing the temperature dependence of R_H is ruled out. The coupling between Cu–O planes increases with the substitution of iron in place of copper. Further, this effect is mild for $x \leqslant 0.07$ and thereafter moderately effective.

(5) The Hall mobility of the positive carriers of all samples increases with decreasing temperature, indicating dominant scattering by phonons.

(6) The experimental values of μ_H are smaller on the lower temperature side than the best possible theoretical values based on e–ph and e–e scattering. An additional scattering mechanism due to the defects that arise from the iron-substituted Cu–O diamond planes is inferred from the temperature variation of the mobility in the sample with $x = 0.3$.

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