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## Effects of iron doping on the transport and magnetic behaviour in $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-y}\text{Fe}_y\text{O}_3$

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**Abstract.** We report on the magnetic and transport measurements in an Fe-doped colossal magnetoresistance compound ( $\text{La}_{0.65}\text{Ca}_{0.35}\text{MnO}_3$ ). Increased spin disorder and a decrease of  $T_c$  with increasing Fe content are evident. We find that the resistivity data above  $T_p$  fits better to a variable range hopping model and the localization length decreases with Fe content. The variations in the critical temperature  $T_c$ , confinement length, magnetic moment and magnetoresistance show a rapid change at about 4–5% Fe. The maximum magnetoresistance is seen to increase consistently with the addition of Fe and increases up to 400% for 8% Fe. The effect of Fe is seen to be consistent with the disruption of the Mn–Mn exchange, possibly due to the formation of magnetic clusters.

### 1. Introduction

The magnetic and resistive behaviour in the colossal magnetoresistance (CMR) compounds [1, 2] is well known to be a sensitive function of the lattice strain produced, for example, by doping on various sites [3–5]. The effects of substitution on the Mn sites by other transition elements is further known [6] to affect the properties due to the changes produced in the average electron concentration and the shifts in the positions of the  $e_g$  and  $t_g$  sub-bands. A number of studies [6–10] have been conducted on the effects of the replacement of Mn by various transition elements, which have included both global and local measurements, such as Mossbauer spectroscopy. The case of Fe is particularly interesting because of the large extent to which it can replace Mn. Ahn *et al* [6] show that the observed decrease in  $T_c$  is consistent with a weakening of the double exchange on the introduction of the Fe. It is apparent, from various studies, that the Fe atom substitutes for Mn in the +3 state and does not take part in the double exchange, thereby leading to a lowering of  $T_c$ . Ogale *et al* [7] showed that their Fe-doped compounds showed a marked decrease in  $T_c$  at a 4% doping level. They related this to the average Fe–Fe separation approaching the size of the charge carriers (polarons) at this concentration. It has also been shown [10, 11] that Fe and Co doping leads to the formation of locally anti-ferromagnetically coupled spins or clusters with localized spin excitations. Several recent studies, including those by neutron scattering, have shown that the onset of the para to ferro transition is accompanied by a divergence of the correlation length, signalling the growth of the infinite correlation and an inhomogenous [12] type of ferromagnetic transition.

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It can therefore be expected that the effect of the Fe doping, which results in the formation of clusters and the suppression of conductivity, would also be reflected in the effective activation energies for hopping transport [13], both above and below the temperature corresponding to the resistivity maximum  $T_p$ . As shown by various studies on CMR compounds [14–17], the resistive behaviour both above and below  $T_p$  can be described in terms of an activated conductivity, albeit with a magnetization-dependent activation barrier for  $T < T_p$ . We have conducted a study of the magnetic and transport effects in a series of Fe-doped CMR compounds of compositions  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-y}\text{Fe}_y\text{O}_3$ , with  $y$  varying as  $0 \leq y \leq 0.1$ , and determined the changes in the correlation lengths, magnetoresistance and magnetization. Our studies are particularly focussed on the effects at low fields,  $H < 15$  kOe, as compared to previous studies. This field range is also of interest from the point of view of potential applications.

## 2. Experiment

### 2.1. Sample preparation

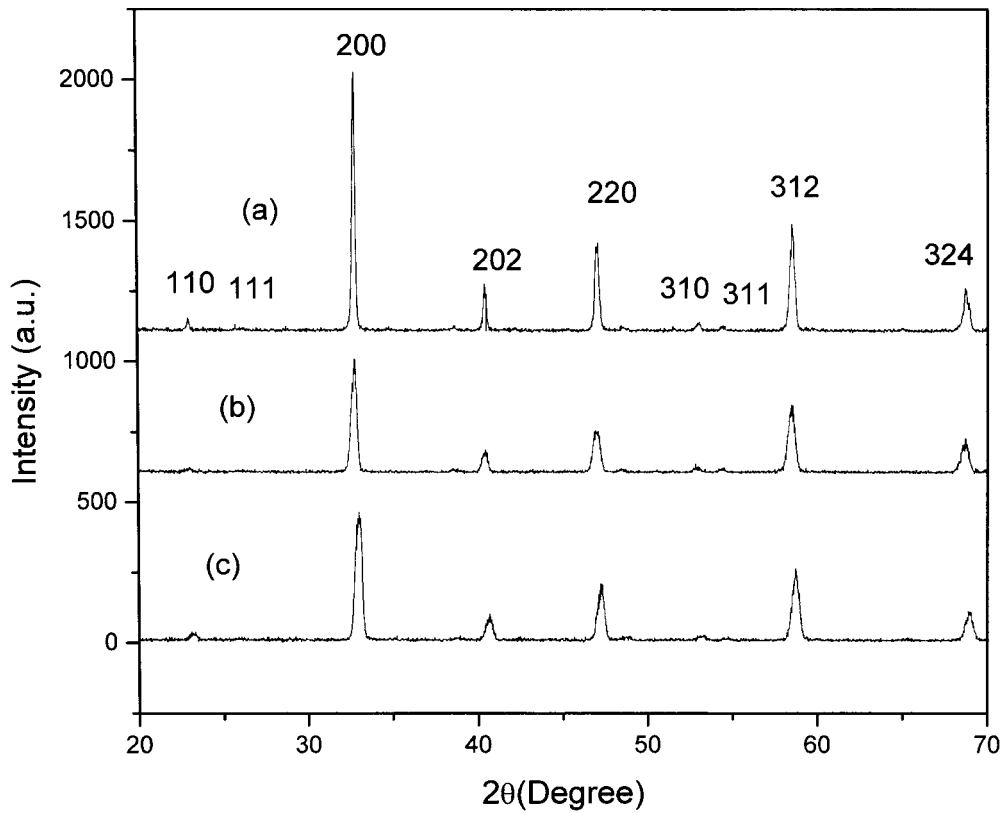
All samples reported in the present study were synthesized by a standard solid-state reaction procedure. Stoichiometric compositions of  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-y}\text{Fe}_y\text{O}_3$  ( $y = 0.0$ – $0.10$ ) were prepared by mixing equimolar amounts of  $\text{La}_2\text{O}_3$ ,  $\text{CaCO}_3$ ,  $\text{Mn}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  (all having greater than 99.9% purity). The powders of these oxides and the carbonate were mixed thoroughly in acetone and were finely ground in an electric grinder for 30 min. After drying, the mixtures were calcined in alumina boats at  $1000^\circ\text{C}$  for 16 h, then cooled to room temperature, reground and again heated at  $1100^\circ\text{C}$  for 17 h. Following cooling to room temperature, they were reground and again heated at  $1200^\circ\text{C}$  for 17 h. After the third heat treatment, the materials were ground to a fine powder and were pressed into pellets of 13 mm diameter and 2 mm thickness under a pressure of 5 tons/inch<sup>2</sup>. These pellets were heated at  $1250^\circ\text{C}$  for 17 h. After this final heat treatment laboratory x-ray diffraction (XRD) measurements were carried out to confirm that single-phase materials had been prepared.

The XRD data were collected by step scanning over the angular range of  $15^\circ \leq 2\theta \leq 70^\circ$  at a step size of  $0.02^\circ$  and counting time of 3 s/step. Figure 1 shows the XRD patterns of the  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-y}\text{Fe}_y\text{O}_3$  collected at room temperature. All peaks were successfully indexed on the basis of a tetragonal unit cell. The lattice parameters, which were obtained using least-squares fitting procedures are  $a = 5.460(2)$  Å and  $c = 7.730(9)$  Å. We do not observe any appreciable change in the lattice parameters with increasing Fe concentration. These results are in agreement with previous findings [6].

### 2.2. Resistivity measurements

Resistivity measurements were performed in a commercial variable temperature liquid nitrogen cryostat. The electrical resistance was measured by using the standard four-probe technique using an air-drying conducting silver paste. The sample temperature was monitored by a calibrated Rh–Fe thermocouple in the range of 77–300 K. The temperature accuracy was 0.05 K. A constant current in the range of  $100 \mu\text{A}$ – $1$  mA was supplied by the current source and voltage across the sample was measured with a digital voltmeter.

Figure 2 shows the dependence of the resistivity as a function of temperature for the  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-y}\text{Fe}_y\text{O}_3$  ( $y = 0.0$ – $0.1$ ) samples. All the samples showed a metal–semiconductor (insulator) transition with a characteristic peak in the resistivity at the transition temperature  $T_p$ . The ferromagnetic transition temperatures  $T_c$  will be shown to be very close to these insulator–metal transition temperatures. It is clearly seen that the transition temperatures

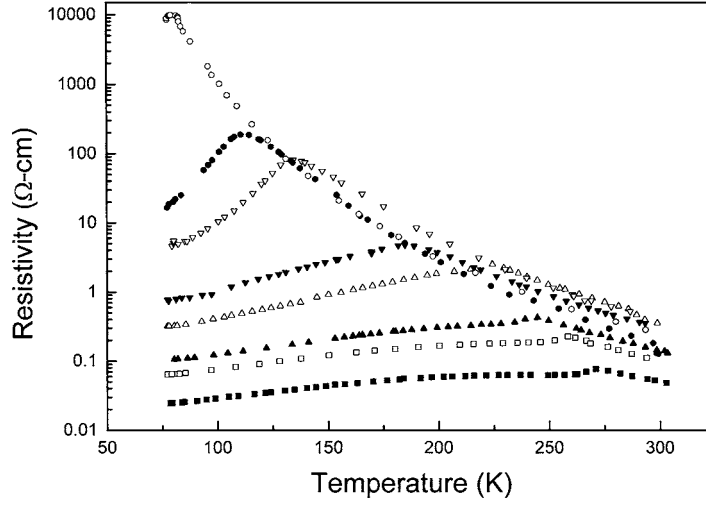


**Figure 1.** XRD patterns for various Fe doping levels in  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-y}\text{Fe}_y\text{O}_3$ : (a)  $y = 0$ , (b)  $y = 0.05$  and (c)  $y = 0.10$ .

are systematically lowered and peak resistivity values increase with the increase in the Fe concentration. This is consistent with the general trend in these systems, where doping with Fe tends to result [6–9] in a weakening of the double exchange interaction, thereby lowering  $T_c$ . This effect is understood to be primarily due to the Fe ions being in the +3 state and hence not participating in the double exchange. The resistivity changes on doping are observed by us to be gradual and linear below 4% Fe concentration, while above this concentration the changes are very large. Note that the resistance is plotted on a logarithmic scale for convenience. We note that the addition of 10% Fe leads to a change of almost six orders of magnitude in the peak resistance. Between 8 and 10% concentration, the peak resistance changes by almost two orders of magnitude while the critical temperature decreases by 30 K. The very pronounced change in the resistivity for  $y > 8\%$  suggests that the disorder exceeds a critical threshold beyond 8%. It is possible that the threshold is connected with the percolation paths in the doped materials.

The two models used to fit the high-temperature resistivity of the manganites are the polaronic model [18, 19] and the variable range hopping model [13]. To facilitate the discussion we shall describe these briefly.

**2.2.1. Polaronic model.** Nearest-neighbour hopping of a small polaron leads to a mobility with a thermally activated form. In the adiabatic regime the charge carrier motion is faster



**Figure 2.** The temperature dependence of the resistivity for  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-y}\text{Fe}_y\text{O}_3$  with  $0 \leq y \leq 0.10$ :  $y = 0.00$  (■);  $y = 0.01$  (□);  $y = 0.02$  (▲);  $y = 0.03$  (△);  $y = 0.05$  (▼);  $y = 0.07$  (▽);  $y = 0.08$  (●) and  $y = 0.10$  (○).

than the lattice vibrations and the resistivity is given by

$$\ln\left(\frac{\rho}{T}\right) = \ln A + \frac{W}{kT} \quad (1)$$

with  $W_p = E_p/2 - t$  as the effective activation energy. ( $E_p$  is the polaron formation energy and  $t$  is the transfer integral [19].) We have used this form for  $T > T_p$  to fit the  $\rho(T)$  behaviour and hence obtain the temperature-independent prefactor  $A$  and the value of  $W$  in (1) for various Fe concentrations.

**2.2.2. Variable range hopping model.** We also fitted the  $\rho(T)$  data for  $T > T_p$  to the variable range hopping model and compared the fit to those obtained from the polaron model. The original expression due to Mott [13]

$$\ln\left(\frac{\rho}{\rho_\alpha}\right) = \left(\frac{T_0}{T}\right)^{1/4} \quad (2)$$

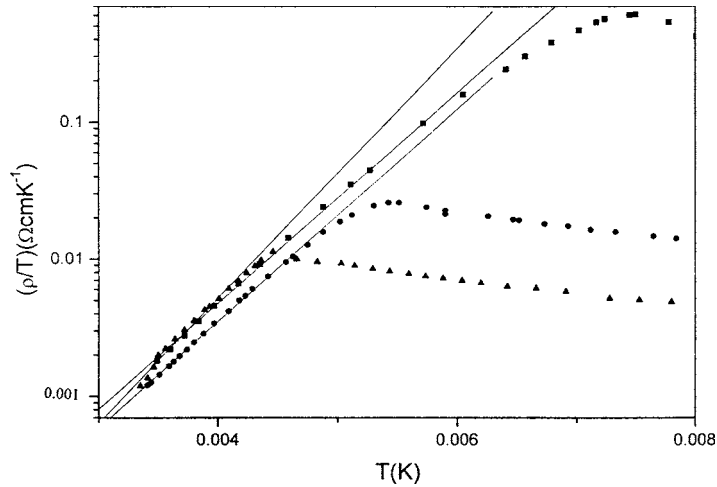
basically describes hopping transport in a system where the carriers are localized by random potential fluctuations and the preferred hopping is between sites lying within a certain range of energies. The above expression for  $\rho(T)$  can be cast in the form

$$\rho = \rho_\alpha \exp[2.06(\alpha^3/N(E)kT)^{1/4}] \quad (3)$$

which is the Mott expression with

$$kT_0 = \frac{18\alpha^3}{N(E)}. \quad (4)$$

In this expression  $\alpha$  is the inverse of the localization length and  $N(E)$  is the density of states. As discussed by Viret *et al* [14, 15] the density of states using the full value of the  $e_g$  electron density leads to an unphysically small value of the confinement length  $1/\alpha \equiv \xi$ . Considering the confinement to be related to the onset of a random *magnetic* potential above  $T_p$ , and also that the hopping probability would be modified by the mutual spin orientations of the two sites,



**Figure 3.** Variation of resistivity with inverse temperature. The lines are fits to the polaronic model for  $T > T_p$ :  $y = 0.03$  (▲),  $y = 0.05$  (●) and  $y = 0.07$  (■).

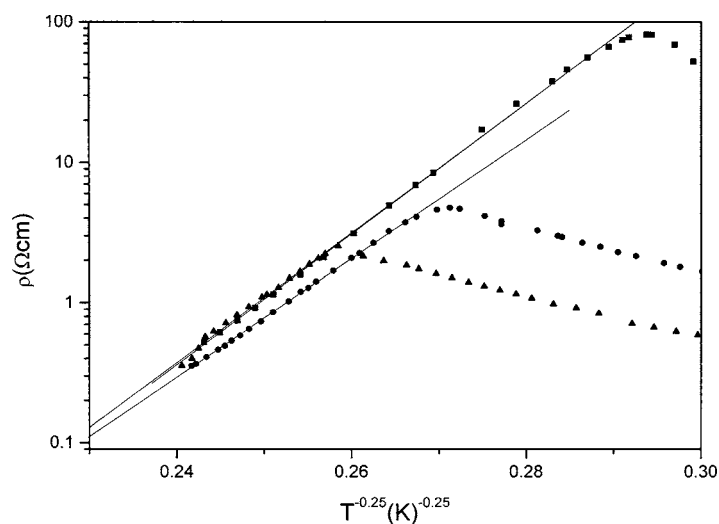
Viret *et al* [14, 15] obtained a reduced effective density of states,  $N(E) = 9 \times 10^{26} \text{ m}^{-3} \text{ eV}^{-1}$ . With these considerations, equation (4) yields

$$kT_0 = 171\alpha^3 U_m v \quad (5)$$

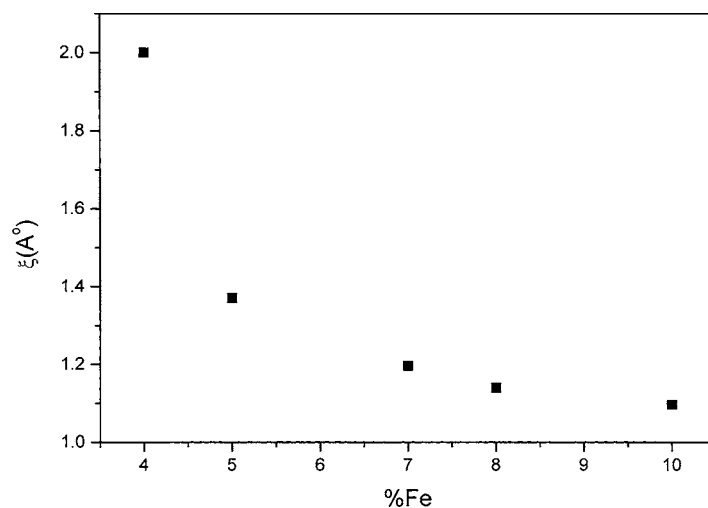
where  $U_m = 2 \text{ eV}$  is the magnitude of magnetic potential and  $v$  is the lattice volume per manganese ion,  $5.7 \times 10^{-29} \text{ m}^3$ . Thus the fit to the variable range model can give  $T_0$  which can be used to obtain the confinement length  $\xi$ .

In figure 3  $\rho/T$  is shown, on a logarithmic scale, as a function of the inverse temperature for some of the compositions ( $y \leq 0.07$ ). The lines are the fits to the polaron model, (2). In figure 4 the resistivity  $\rho$  is shown on a logarithmic scale as a function of  $T^{-0.25}$  for the same sample. The lines are fits to the variable range hopping model, (3).

Since we did not have a sufficient number of data points at  $T \gg T_p$  for the low-concentration samples (for which  $T_p$  is high), the fitting was performed for  $y \geq 3\%$  Fe. We find that the variable range hopping model shows a good fit to the data almost all the way down to the peak, while the fit to the polaronic model is slightly worse, particularly close to the peak. This can be seen from a comparison of the fits in figures 3 and 4 and is clearly evident for the 7% sample. This suggests that the conduction above  $T_p$  is more readily describable, as in the variable range hopping model, by a hopping of the charge carriers localized by random potential fluctuations. As discussed above, the resistivity data was used to obtain the values of  $T_0$  from the fit. Using equation (5) and the density of state values described above, the  $T_0$  values were used to obtain the localization length  $\xi$  for the different Fe concentrations. Figure 5 shows the variation of  $\xi$  as a function of the Fe concentration above 4%. It can be seen that  $\xi$  decreases as the Fe concentration is increased. Although we have obtained  $T_0$  and  $\xi$  values for all samples, for samples having an Fe concentration less than 4%, however, these values may not be reliable due to the limited temperature range available for the fit above the transition temperature. We find that the localization length  $\xi$  decreases from 2 Å at 4% Fe, to 1.1 Å at 10% Fe. Using the same method Viret *et al* [15] obtained 4.5 Å for La–SrMnO<sub>3</sub> and 0.8 Å for Pr–PbMnO<sub>3</sub>, while Zeise *et al* [16] obtained between 0.3 and 0.7 Å for their thin-film samples. We note from our data figure 5 that there is a noticeably large change in the localization length at a 5% Fe concentration, consistent with the observation of Ogale *et al* [7]



**Figure 4.** Resistivity of  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-y}\text{Fe}_y\text{O}_3$  with  $y = 0.03$  ( $\blacktriangle$ ),  $y = 0.05$  ( $\bullet$ ) and  $y = 0.07$  ( $\blacksquare$ ) against  $T^{-0.25}$ . The lines are fits to the variable range hopping model.

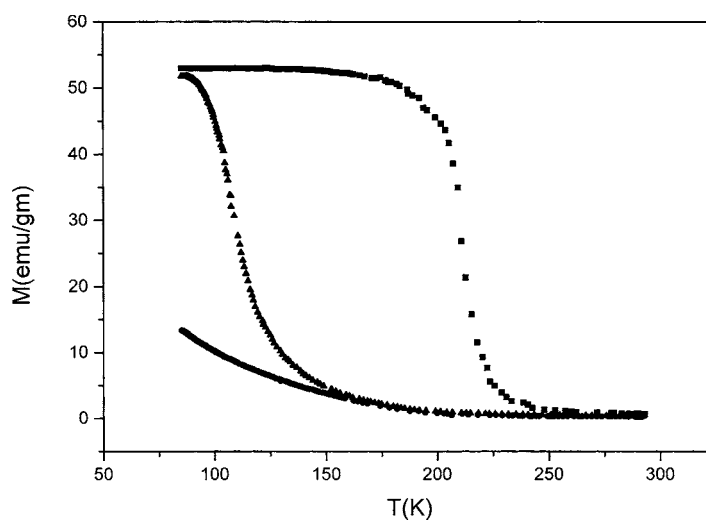


**Figure 5.** Variation of the confinement length  $\xi = 1/\alpha$  obtained from the fits to the variable range hopping model as a function of the Fe content.

who point out that the conduction decreases rapidly at about 4%, where the Fe–Fe separation begins to be comparable to the size of the charge carriers.

### 2.3. Magnetization measurements

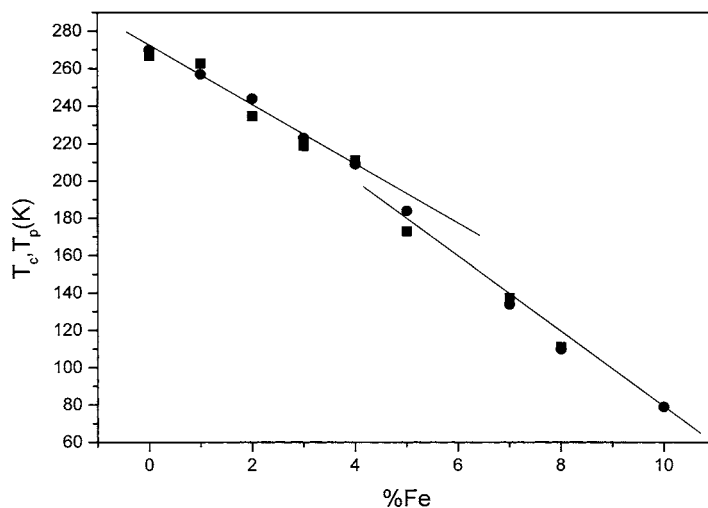
The magnetization as a function of temperature ( $T > 84$  K) was studied for all the samples, with a 1 kOe field applied during cooling. All the samples except the sample with  $y = 0.1$  (10% Fe) showed a transition to the ferromagnetic state in this temperature range. The  $y = 0.1$  sample had a magnetization which rapidly increased as the temperature was decreased below  $T \sim 175$  K without however approaching saturation. The 8% Fe sample meanwhile shows a



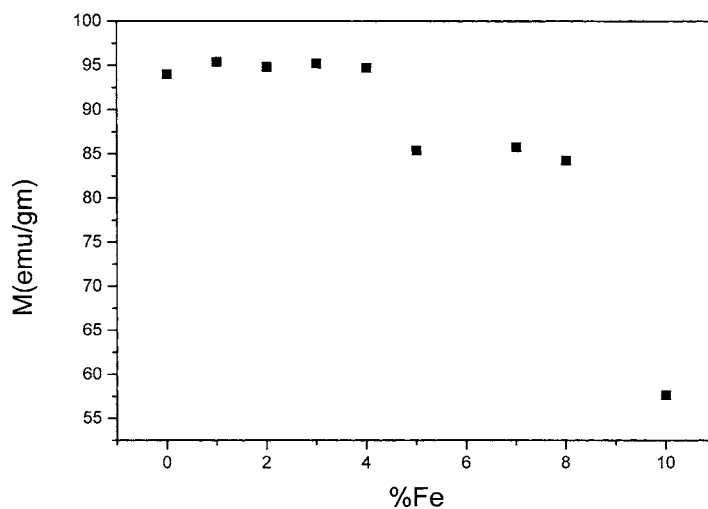
**Figure 6.** Variation of the magnetic moment with temperature for the 4, 8 and 10% Fe-doped samples with  $y = 0.04$  (■),  $y = 0.08$  (▲) and  $y = 0.10$  (●).

magnetization approaching saturation at  $T \sim 90$  K. A typical temperature dependence of the magnetization is shown in the figure 6 for the 4, 8 and 10% samples. The critical temperature was defined as the midpoint (50% point) of the transition, i.e. where the magnetization attains 50% of the low-temperature, approximately saturated value. The transition width  $\Delta T_c$  was defined as the region in which the magnetization changed from 10 to 90% of its full value. With this definition of the transition width, it was seen to change from 22 to 46 K over the composition range from 0 to 8%. The increasing width of the transition is consistent with an enhanced spin disorder due to Fe doping. For the critical temperature  $T_c$ , as defined above, its variation as a function of the Fe concentration is shown in figure 7. Also shown for comparison are the  $T_p$  values obtained from the resistivity peak. We note that the variation of  $T_c$  and  $T_p$  with the Fe content has a change in slope above 4% Fe, but this change is certainly not quite as sudden as in the case of Ogale *et al* [7]. We have already seen that the resistive data shows a marked change in the values of various parameters at this concentration. On a similar note we find that the high-field moment also shows a very abrupt decrease at this concentration. This can be seen in figure 8, where the moment at 77 K and 15 kOe is shown for various compositions. It is clear from these data that the high-field moment, which is relatively constant for  $y \leq 0.04$ , decreases abruptly for the 5% composition. Beyond this composition there is again a small change with composition till  $y = 0.10$ . For this latter composition the sample, however, has not completed the ferromagnetic transition at 77 K. Thus both the 4 and 8% concentrations appear to mark the onset of large changes in the magnetic and resistive behaviour. All the compositions display negligible hysteresis. While all the  $y < 0.1$  samples show a very rapid rise to saturation with increasing field, the  $y = 0.1$  composition depicts a much slower increase with field and a pronounced curvature up to our highest field of 15 kOe. While increasing antiferromagnetic correlations with increasing Mn are consistent with the continuous decrease of  $T_c$  and the increase of resistance, the abrupt changes in the magnetic and resistive features at 4 and 8% would indicate that for these compositions there are some additional factors affecting the magnetic and resistive behaviour. Local probes such as Mossbauer or neutron scattering would be required to address this further.





**Figure 7.** Variation of  $T_p$  and  $T_c$  with the Fe concentration for, respectively, resistively (●) and magnetically (■) obtained critical temperatures. The lines are guides to the eye. The change in slope above 4% Fe is apparent.



**Figure 8.** Variation of magnetic moment (77 K) against the Fe concentration at 15 kOe for  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-y}\text{Fe}_y\text{O}_3$ . The sharp drop at  $y = 0.05$  is evident.

#### 2.4. Magnetoresistance studies

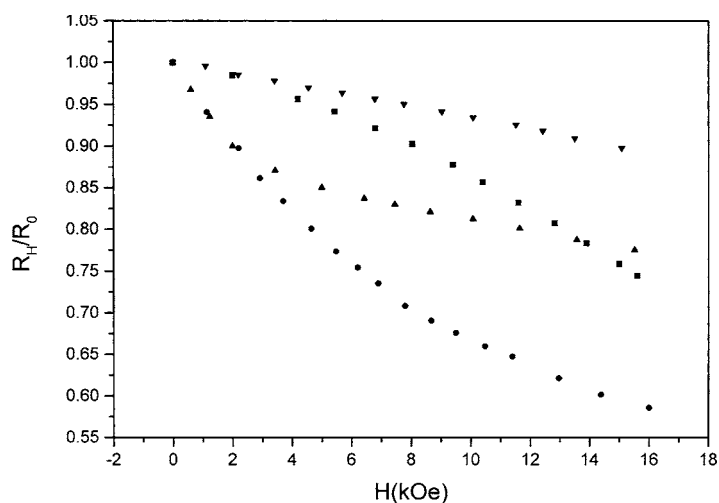
We performed magnetoresistance studies on most of the compositions in the range Fe = 0–10% at various temperatures in the field range of 0–15 kOe. We were specifically interested in determining whether the abrupt changes that appear at  $y > 0.04$  are also exhibited in the magnetoresistance. Previous studies [8], in contrast, have usually concentrated on high-field effects and have not addressed this latter point. Typical magnetoresistance behaviour is shown in figure 9 for the 4% composition for the temperatures 242, 223, 209 and 104 K. The resistance values have been normalized to the zero-field values at each temperature. The critical

temperature of this sample was 209 K. The general features of magnetoresistance in this series of compounds are evident in the 4% material. These include maximum magnetoresistance effects at  $T = T_c$ . Furthermore, there are significant effects both above as well as below  $T_c$ . While the effects at and above  $T_c$  may be associated with the field suppressing the spin fluctuations and hence leading to the decrease of the resistance, the effects well below  $T_c$ , for example at 104 K, can originate from both intergrain and intra-grain sources. If, as expected in the doped compounds, there were intra-grain spin disorder below  $T_c$  as well, the field would act to decrease it. Furthermore, for  $T \ll T_c$  the spin disorder within the grain boundaries is also understood to be a contributing factor to the negative magnetoresistance in these materials [20, 21] in general. To compare the magnetoresistance effects in the various compositions, we show in figure 10 a plot of the percentage magnetoresistance as a function of the Fe composition at the respective critical temperatures, (except for  $y = 0.1$  for which the data are at  $T = 88$  K, which is above its  $T_c$ ). The percentage magnetoresistance is defined as  $[R_{(0)} - R_{(H)}]/R_{(H)} \times 100$ , where  $R_{(0)}$  is the resistance at zero field while  $R_{(H)}$  is the resistance for an applied field of 15 kOe and the same temperature. It is clear that with increasing Fe content the magnetoresistance systematically increases. In [7] the authors do not report significant changes in the maximum effect up to  $y = 0.0475$ , i.e. 4.75% Fe, and  $H = 1$  T, for their 25% Ca-doped samples. In our case, i.e. with 35% Ca, we find pronounced changes even at low Fe concentrations. It is possible that this difference for low Fe compositions arises from the fact that we start from the optimum Ca-doped sample, while in their case they started from a significantly lower Ca composition. In the undoped sample the maximum magnetoresistance values are 30%, while for 2% Fe it is 47%. Similarly the maximum magnetoresistances for the 4, 7 and 8% Fe doped samples were 71, 249 and 405%, respectively. For the 10% Fe sample, the magnetoresistance effect was only 42% and is understandably low compared to the other compositions because the temperature ( $T = 88$  K) was above  $T_c$ . There again we notice the very pronounced change occurring at  $y > 4\%$ . The very large field-induced decrease in the resistance is consistent with the increasing spin disorder with Fe doping. On the application of the magnetic field this disorder decreases drastically, and consequently the resistance also decreases. It thus appears that the spin disorder due to high Fe content results in the locally ordered clusters, most probably of an antiferromagnetic alignment. At  $T_c$ , where the spin correlations are weak, these clusters appear to break readily, even in moderate fields. These spins then become part of the main ferromagnetic system. This extension of the ferromagnetic correlations of course facilitates the conduction.

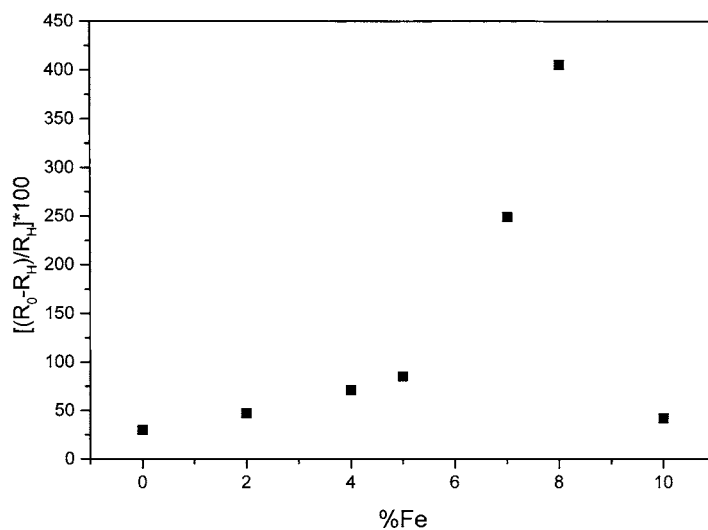
The very large magnetoresistive effects at high Fe doping are consistent with the generally observed trend in the CMR compounds where increasing disorder and narrower electron bandwidths are accompanied by stronger magnetoresistive effects. The abrupt change at about a 5% Fe content again suggests that when the average Fe–Fe separation become comparable to or smaller than the typical polaron size, the effects of spin alignment on the conduction are very pronounced.

### 3. Conclusions

Our study of the transport and magnetic behaviour of the Fe-doped CMR compounds are consistent with previous studies, in the sense that they show a general decrease of  $T_c$  with an increasing Fe content. We find that the variable range hopping model gives reasonable values of the parameters and appears to fit the data better than the polaronic model. The variable range hopping model used with the corrections suggested by Viret *et al* yields values of the confinement length that show a systematic decrease with Fe content, which is again very strong at 4%. The behaviour of the confinement length with concentration supports the observation



**Figure 9.** Magnetic field dependence of the resistance for  $y = 0.04$  at temperatures of 242 K ( $\blacktriangledown$ ); 223 K ( $\blacksquare$ ); 209 K ( $\bullet$ ) and 104 K ( $\blacktriangle$ ).  $T_c = 209$  K. The resistance values are normalized to corresponding zero-field values at each temperature.



**Figure 10.** The percentage magnetoresistance  $(R_{(0)} - R_{(H)})/R_{(H)} \times 100$  for various Fe concentrations measured at 15 kOe and the corresponding critical temperature  $T_c$ . For 10% Fe the measurement is at  $T = 88$  K, above  $T_c$ . Large effects are evident above 5% Fe.

that the conduction and magnetic effects undergo a sudden change at this concentration, which has been suggested to be due to the closeness of the Fe–Fe separation with the typical polaronic size in these materials. In contrast to a previous report on Fe doping effects in CMR compounds with a slightly lower calcium content, we find that the magnetoresistance effect is sensitive to the Fe content. In particular, there is a pronounced increase at the concentration where the  $T_c$  and high-field moment also show abrupt changes. Effects as large as 400% are obtained. These may be associated with the increasing disorder in the bond angles, the consequent difficulty of hopping and the narrowing of the electron bandwidth with increased doping. However,

we note that the relationship between clustering effects in the CMR compounds and the large magnetoresistance has been suggested by several authors [10–12, 22]. It is quite possible that the large magnetoresistance effect in the compounds with a higher Fe concentration is a consequence of the tendency for clustering due to the antiferromagnetic interaction of Fe with its neighbours. In view of the reports on these doped materials regarding the formation of spin clusters, it seems reasonable to argue that the effect of the field is to decrease the spin disorder, both above and below  $T_c$ , thereby increasing the spin correlations and the conductivity. At  $T_c$ , where the correlations are not fully established, the field apparently helps stabilize the ferromagnetic correlations and leads to large resistivity changes in the doped materials.

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