Temperature and magnetic-field dependence of the resistivity of carbon-black polymer composites

P. Mandal, A. Neumann, A. G. M. Jansen, and P. Wyder
Grenoble High Magnetic Field Laboratory, Max-Planck-Institut für Festkörperforschung and Centre National de la Recherche Scientifique, Boîte Postale 166, F-38042 Grenoble Cedex 9, France

R. Deltour
Université Libre de Bruxelles, Service de Physique des Solides, Code Postal 233, Boulevard du Triomphe, B-1050 Bruxelles, Belgium
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The dc electrical resistance of carbon-black polymer composites has been studied over a wide temperature range from 300 to 0.35 K and in high magnetic fields up to 20 T for different carbon concentrations above the percolation threshold. The temperature dependence of the resistivity of all samples follows a Mott variable-range-hopping dependence like $\ln\rho \sim T^{-n}$ with $n > 1/2$ over the whole temperature range. This temperature dependence with $n > 1/2$ may be understood on the basis of the superlocalization of electronic states in the fractal structure of the carbon-black polymer. At high temperatures where the hopping distance is comparable to the localization length, the magnetoresistance is small and negative, which may be accounted for by the suppression of quantum interference effects. The large and positive magnetoresistance at low temperatures where the hopping distance is larger than the localization length may be due to the shrinkage of impurity orbitals in the magnetic field. [S0163-1829(97)01002-3]

I. INTRODUCTION

For understanding of the electrical transport in disordered systems a large variety of phenomena, such as localization effects, hopping transport, and percolation, play an important role.[1–3] The occurrence of a certain phenomenon depends on the nature and strength of the disorder in the studied system. Usually, disorder is introduced on a microscopic level either by impurity doping or by disrupting the crystalline structure of the material. Another well-known technique is to make an intrinsically insulating material, such as a polymer, conducting by incorporating in its matrix a dispersion of macroscopic conducting particles like carbon black (CB) or fine metallic particles.[4] These composites show a drastic change in electrical conductivity of many orders of magnitude with the variation of the conducting particle density in the vicinity of the critical concentration. This gives an excellent opportunity to study percolation phenomena in a binary system.

Recently, different groups have investigated the variable-range-hopping (VRH) phenomenon in carbon-black polymer composites.[5,6] In the conventional VRH theory[3,7,8] the temperature dependence of the resistivity follows an expression of the form $\rho(T) \sim \exp(T_0/T)^n$. In three-dimensional (3D) systems such as ours $n = 1/4$ and the value of T_0 is usually large compared to the measured temperature range.[3] In many systems where electron-electron correlation plays an important role, a so-called Coulomb gap opens in the density of states, and $n = 1/2$ is observed at low temperatures.[3] van der Putten *et al.*[6] measured the temperature dependence of the dc conductivity of CB polymers as a function of carbon concentration in the temperature range 4–300 K and observed VRH conduction with $n \sim 2/3$ and a very small value of $T_0 \sim 112$ K for all the samples irrespective of carbon concentration and its conductivity. They interpreted this unusual behavior of the transport phenomena in terms of the superlocalization of the electronic states in the presumably fractal CB polymer, and deduced that the wave function of the electronic states at the Fermi level decays with distance x as $\exp[-(x/L)^{\zeta}]$ where $\zeta = 1.94$ and L is the localization length, instead of purely exponentially ($\zeta = 1$). However, Aharony *et al.*[9] described these experimental data with the Mott VRH expression including a temperature-dependent prefactor and obtained a very large value of T_0 and a comparatively smaller value of n. They claimed that the transport phenomena in CB polymers are not different from usual VRH conduction and suggested measurements at temperatures below 4.2 K where a significant difference between the two interpretations would be expected. The smaller value of T_0 compared to the measured temperature range also prevents a decisive interpretation of the experimental data of Ref. 6.

In order to elucidate the transport mechanism in CB polymers we have measured the resistance and magnetoresistance as a function of carbon concentration down to very low temperatures.

II. SAMPLE PREPARATION AND EXPERIMENTAL TECHNIQUES

Two batches of CB polymer composites were prepared with two different kinds of CB: Ketjenblack EC 300N and EC 300J from AKZO Chemie Company, Arnhem, The Netherlands. Hereafter, we refer to these two groups of CB polymers as batch I and batch II, respectively. The conducting CB particles contain graphitelike layers of carbon atoms and their radius is about 20–25 nm. All the samples were prepared by the solution technique. In this method, polystyrene is dissolved in a solvent (xylene). A known amount of well-dispersed CB is added to the solution and the mixture is stirred by means of a magnetic agitator for more than 24 h in

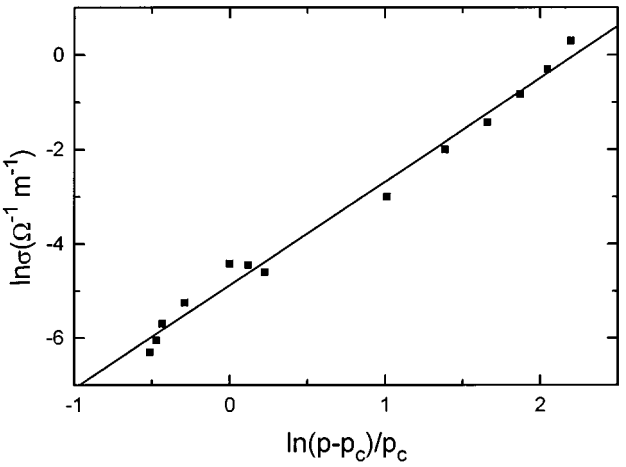

FIG. 1. Logarithm of the room-temperature dc conductivity σ as a function of the logarithm of the carbon particle concentration for batch I samples.

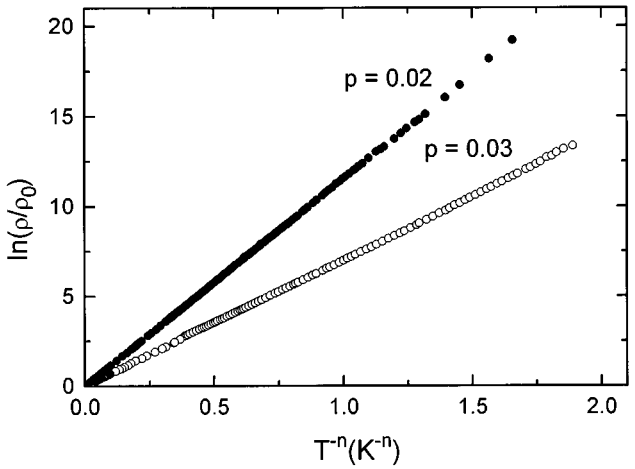

FIG. 2. The temperature dependence of the resistivity of two samples from batches I and II, plotted in the form $\ln[\rho(T)/\rho_0]$ vs T^{-n}. $n=0.79$ for $p=0.02$ and $n=0.65$ for $p=0.03$ samples.

order to obtain a homogeneous distribution of the CB inside the polymer matrix. The solution is then deposited on glass slides and the solvent is allowed to evaporate very slowly in a saturated xylene atmosphere. Finally, the samples are cut in rectangular form from the films that have a thickness of 0.2 – 0.3 mm.

For the resistance measurements we used samples with dimensions of $\sim 15\times 10$ mm^2. Four electrical contacts were made by fixing copper wire by means of the conducting carbon-paint Electrodag. Resistance measurements were done in two steps: in the temperature range from 300 to 4.2 K using a ^{4}He cryostat and below 4.2 K using a ^{3}He cryostat. We used the Keithley 196 and 642 dc electrometers and 220 current source. The very high input impedance of these meters allowed us to measure the resistances of batch I and batch II samples down to 0.5 K and 0.35 K, respectively.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Temperature dependence of the resistivity

In a percolation network, a finite value of the conductivity will be observed when the concentration p of the conducting particles exceeds a threshold value p_c. Above this critical concentration conductivity follows a scaling behavior of the form[3] $\sigma(p)=\sigma_0[(p-p_c)/p_c]^t$, where σ_0 is a constant and the exponent t depends on the dimensionality and the kind of the percolation network of the system. In Fig. 1 we have plotted the logarithm of the conductivity ($\ln\sigma$) versus $\ln[(p-p_c)/p_c]$ for the first batch of our samples. From the fit we find $t=2.2$ and $p_c=0.002$. The value of the critical exponent (t) is slightly larger than the theoretical value[3] for a 3D system ($t=1.6$–2.0 depending on the network geometry) but close to the values reported by others.[5,6,10] The small value of p_c suggests that the solution technique is useful to obtain a small percolation threshold.

The temperature dependence of the resistivity of all the samples has been analyzed using the Mott VRH expression of the form

$$\rho(T)=\rho_0\exp(T_0/T)^n, \qquad (1)$$

where ρ_0, T_0, and n are parameters. The exponent n determines the nature of the conduction mechanism in the system.[3] In Fig. 2 we have plotted $\ln[\rho(T)/\rho_0]$ versus T^{-n} for two samples from batches I and II to illustrate the temperature dependence of the resistivity of our samples. The values of the parameters T_0 and n obtained from the best fit using Eq. (1) are presented in Table I. From Table I it is clear that neither T_0 nor n show any variation with the carbon concentration. All the samples of batch I may be fitted well with $n\simeq 0.78$ and $T_0\simeq 27$ K, and for batch II these values are 0.65 and 19 K, respectively. The large effective temperature range (T^{-n}) and the resistivity variation over six to seven decades in this range allow an accurate determination of n.

The above values of n for CB polymers are too large compared to the usual VRH theory, where $n=1/4$ for a 3D system or $n=1/2$ when taking into account a Coulomb gap in the density of states. Moreover, in the conventional theory the parameter T_0 depends on the localization length and would increase with disorder.[11,12] Such behavior has not been observed in our CB composites. We did not see any variation of T_0 with carbon concentration. In the following we will discuss several theoretical models to shed some light

TABLE I. The values of parameters T_0 and n obtained from the best-fit results using Eq. (1).

Batch	p	$T_0(K)$	n
I	0.0050	26	0.783
	0.0100	28	0.777
	0.0075	26	0.775
	0.0125	27	0.780
	0.0150	28	0.767
	0.0175	27	0.780
	0.0200	25	0.792
II	0.0250	20	0.66
	0.0300	23	0.65
	0.0350	17	0.64
	0.0400	18	0.64
	0.0500	16	0.65

on the anomalous behavior of T_0 and the value of n in view of variable-range-hopping transport.

A temperature dependence of the resistivity with $n>1/2$ and a small value of T_0 above 4.2 K have been reported by others for CB systems.[5,6] The authors of Ref. 6 interpreted their results by assuming superlocalization of the electronic states in the fractal CB polymer. Levy and Souillard[13] predicted that on a fractal network the wave function of both vibrational and electronic states at the Fermi level decays at a distance x as $\exp[-(x/L)^{\zeta}]$, where L is the localization length and $\zeta>1$, instead of purely exponentially ($\zeta=1$) as in the case of Anderson localization. Based on this prediction, Deutscher, Levy and Souillard[14] extended Mott's VRH model to a fractal network to explain the temperature dependence of the hopping conductivity with n close to 1/2 in the absence of a Coulomb interaction and T_0 independent of p. Assuming the general form for the density of states, $N(E)=BE^k$, van der Putten *et al.*[6] derived expressions for the different energy and length scales and a more general form for the exponent n for hopping conduction between superlocalized electronic states:

$$k_B T_0=[(k+1)c/(BV_L)]^{1/(k+1)}n^{-1}(1-n)^{1-1/n}, \quad (2)$$

$$(R_h/L)^{\zeta}=(1-n)(T_0/T)^n, \quad (3)$$

and

$$n=[1+D/\zeta(k+1)]^{-1}, \quad (4)$$

where B, c and k are constants, D is the fractal dimension of the system, V_L is the volume of a localized state, and R_h is the typical hopping distance. The Mott and Efros-Shklovskii expressions for hopping conductivity may be recovered from these equations in special cases. Below 4.2 K, $(R_h/L)^{\zeta}$ for our samples is in the range 1–5. This implies that the Mott criterion for VRH conduction is satisfied at low temperatures.

For temperatures above 4.2 K the value of $(R_h/L)^{\zeta}$ is smaller than 1, implying that a hopping mechanism is no longer applicable. Such small a hopping distance has also been found at higher temperatures for other studies on carbon-black polymer composites[5,6] and even for systems showing classical VRH behavior like CdSe.[15] In the region $R_h/L\simeq 1$ one could expect a change in the temperature dependence of the resistivity with, e.g., a metallic type of temperature dependence ($d\rho/dT>0$) or (weak-)localization behavior ($d\rho/dT<0$). For our data we did not observe a change in the temperature dependence in the whole temperature range from 400 mK until 300 K. The variation with temperature by orders of magnitude excludes also a description in terms of weak-localization theory using power-law dependences reflecting the temperature-dependent inelastic scattering. Although the VRH criterion does not hold for the whole temperature region, it is surprising that the whole temperature region studied can be described with an exponential temperature dependence related to the VRH theory.

The localization lengths estimated from Eq. (2) using our experimental values of T_0 and n, and with $k=1$, $c=1$, $B=3.7\times 10^{21}$ cm^{-3} eV^{-2} (as mentioned in Ref. 6 for carbon black), are about 100 and 120 nm for batches I and II, respectively. It is known that at length scales smaller than the percolation length ξ_p the regions accessible to the electrons are fractal.[14] This implies that both the localization length and hopping distance should be smaller than ξ_p. The precise determination of ξ_p is very difficult, but if we assume $\xi_0\sim$ size of a CB particle in the expression of the scaling theory,[3] $\xi_p=\xi_0(p-p_c)^{-\gamma}$ with $\gamma\sim 0.9$, then ξ_p is at least one order of magnitude larger than R_h and L for all the samples. As R_h increases with the decrease of temperature [Eq. (3)], it is expected that at low temperatures where $R_h\sim\xi_p$ the electron does not ''feel'' any longer that the medium is fractal. In such cases the wave function will decay exponentially as usual and a crossover to conventional Mott VRH conductivity with $n\leq 1/2$ will be seen. Even for the sample with the highest carbon concentration we did not observe any crossover to a smaller value of n at low temperatures. This also supports the contention that ξ_p remains larger than R_h down to the lowest temperature we have used.

We now discuss the observed value of n in view of the theoretical predictions for superlocalized electronic states. Using the value of n in Eq. (1), the ratio D/ζ can be determined. In this way, the different values of n for the two different kinds of CB particles can be related to the fractal dimension D and the superlocalization exponent ζ which will depend on the particle structure and distribution. Different values for n have also been found for different preparation methods of the polymer composites.[5] However, according to the Alexander-Orbach conjecture,[16] $D/\zeta=4/3$ for a percolation network in all dimensions. Using the limiting values $0\leq k\leq 1$, we obtain, from the values of n, $0.28<D/\zeta<0.56$ for batch I samples and $0.54<D/\zeta<1.1$ for batch II samples. Only the limiting $k=1$ value for batch II approaches the theoretical conjecture. From the estimation of the Coulomb energy due to the interaction between charged particles van der Putten *et al.* suggested a possible crossover from hopping dominated by the density of states ($k=1$) to Coulomb-dominated hopping at low temperatures. At low temperatures where the Coulomb effect is dominant and a pseudogap is induced at the density of states, a unique relation $n=\zeta/(\zeta+1)$ is determined,[6,9] which is a generalization of the Efros-Shklovskii expression for a fractal network. For a Coulomb gap, $\zeta=1.34$ which yields $n=0.57$.[9] Though this value of n is close to the observed exponent for batch II samples, it is certainly smaller than the one we obtained for the other samples.

Aharony *et al.*[9] suggested an inclusion of a temperature-dependent prefactor in the VRH expression, i.e., $\rho(T)=\rho_0(T/T_0)^s\exp(T_0/T)^n$. They claimed that the resistivity data of Ref. 6 and others where a very small value of T_0 has been obtained can be analyzed using this expression with a very large value of T_0 and a relatively smaller value of n. We also tried to fit our resistivity data with this expression and observed that s is very close to zero with a very small change in T_0 and n compared with those suggested by Aharony *et al.*[9] The fits become progressively worse and s takes an unphysically large value when we try to analyze our data with a fixed and lower value of n. A strong deviation between experimental data and the above expression in the low-temperature region as suggested in Ref. 9 is also observed. Moreover, this expression predicts a minimum in $\rho(T)$ at $T=T_0(n/s)^{1/n}$ which has not been observed in our samples. The suggested fit[9] with $n=0.57$ for a Coulomb gap

is somewhat surprising because at high temperatures it is very hard to realize that the Coulomb interaction will be dominant.

Sheng and Klafter[17] deduced the temperature dependence of the conductivity for high-resistivity granular-disordered systems based on the critical path method introduced by Ambegaokar *et al.*[18] near the percolation threshold in conducting composites. In this model, the density of states is not constant but obeys a power law $N(E)=N_0+\alpha E^{\beta}$, where the constants α and β are determined by the size distribution of the particles in the composites. It has been shown by Sheng and Klafter[17] that this energy-dependent average density of states leads to a well-defined value for the exponent in the temperature-dependent resistivity with $n=(1+\beta)/(2+\beta)$ at high temperatures. According to this theory $n\sim 1/2$ is obeyed over a large temperature range with a possible crossover to $n=1/4$ at low temperatures and $1/2<n<1$ at high temperatures. However, in our CB polymers we did not see the above temperature dependence for $\rho(T)$ and the crossover as predicted by Sheng and Klafter for granular systems. Furthermore, in this theory, T_0 is strongly concentration dependent in contrast to our findings. The fluctuation-induced tunneling model specifically proposed for CB polymers by Sheng *et al.*[19] is also not able to explain the temperature dependence of the resistivity of our samples. In this model, the resistivity is given by $\rho(T)=\rho_0\exp[T_0/(T_1+T)]$. Thus the resistivity is almost temperature independent at low temperatures below the characteristic temperature T_1 (~ 1 K) and behaves as predicted by thermal activation in the high-temperature region ($T\gg T_1$).

B. Magnetoresistance

We have measured the magnetoresistance $(\Delta\rho(H)/\rho(0)=[\rho(H)-\rho(0)]/\rho(0))$ for magnetic fields up to 20 T for the samples of batch I. At temperatures above 8 K the magnetoresistance is very small and negative while it is large and positive below 4.2 K. Also, at a given temperature, both magnitude and field variation of $\Delta\rho(H)/\rho(0)$ are the same for all the samples and independent of the relative direction of current and magnetic field. Thus the magnetoresistance is independent of the density of conducting particles, as are T_0 and n.

In Fig. 3 we show the magnetoresistance data for a $p=0.01$ sample. At 20 T the magnetoresistance increases from 38% to 102% as the temperature decreases from 4.2 to 1.45 K. From the magnetoresistance vs magnetic field curves we define three regions of low, intermediate, and high magnetic field. At low fields, $\ln[\rho(H)/\rho(0)]$ is proportional to H^2. In the intermediate-field range, the magnetoresistance is approximately linear in H. The dependence of the magnetoresistance on H in the high-field region is much weaker (sublinear) and reaches saturation at the highest fields and the lowest temperatures. The field range of these regions is very sensitive to temperature and decreases as the temperature decreases. At 4.2 K we observed the H^2 behavior up to $H\sim 6$ T, while at 1.45 K it is seen only below 2 T. At 4.2 K the crossover from the H^2 to the H dependence occurs at about 7 T and the linear behavior remains up to fields as high as 16 T. The intermediate-field range also gets narrower with decreasing temperature.

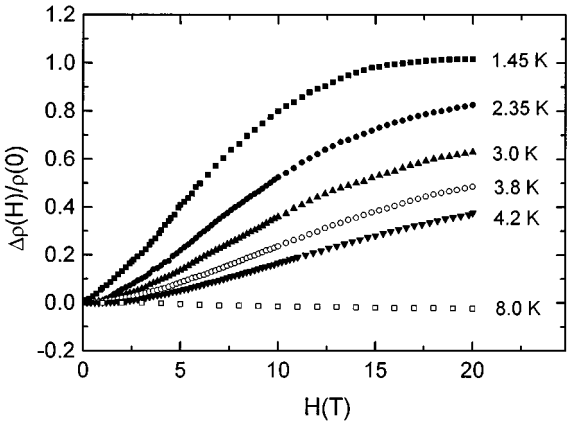

FIG. 3. The magnetoresistance of a $p=0.01$ sample (batch I) as a function of H at various temperatures.

Jiang *et al.*.[20] and Iwasaki *et al.*[21] have reported magnetoresistance measurements on insulating $Y_{1-x}Pr_xBa_2Cu_3O_7$ single crystals and thin films in which 3D VRH ($n=1/4$) is observed. At low temperatures they observed a large positive magnetoresistance but a small negative magnetoresistance at higher temperatures. Both the temperature and magnetic-field dependence of $\rho(H)/\rho(0)$ of their samples is very similar to our results. The magnitude of $\rho(H)/\rho(0)$ of our samples are also comparable to the magnetoresistance of the Pr-doped $YBa_2Cu_3O_7$ samples.

The effect of the magnetic field on the carrier transport in the VRH regime where the electrons are strongly localized is not yet fully developed. The simplest picture is that the application of a magnetic field causes the impurity orbitals to shrink, leading to a decrease in the tunneling probability, and as a consequence a large positive magnetoresistance is expected. This has been observed in many semiconductors where a resistivity increase of several decades with magnetic field is seen.[3] At low temperatures, where the hop distance is much larger than the average separation between the impurities the electron can be scattered by several impurities before arriving at the destination impurity site. The overall tunneling amplitude which determines the conductivity is then obtained from the sum over all possible trajectories between the initial and final sites. Since the contribution of each such trajectory decays exponentially with distance, the shortest or forward scattering will dominate the sum. In such cases, the contribution from backscattering paths may be neglected. The quadratic dependence of the magnetoresistance on H in the weak-field region is consistent with this picture as has been shown in Ref. 3 and 22 in the strongly localized regime where $R_h\gg L$. In contrast, at high temperatures where the hopping length is comparable to the localization length the system may not be considered to be deep in the strongly localized region. In such cases, backscattering phenomena become important and the weak-localization regime sets in.[1,23] The observed small negative magnetoresistance at high temperatures in our samples where $L\sim R_h$ is consistent with the weak-localization picture.

We now analyze the positive magnetoresistance at low temperatures. The shrinkage of impurity orbitals in a magnetic field means a decrease of the localization length. This may be estimated from the variation of T_0 with magnetic

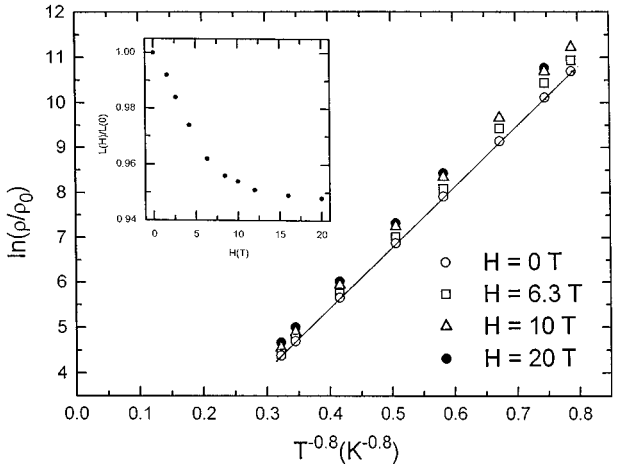

FIG. 4. The plot of $\ln\rho$ vs $T^{-0.8}$ for a $p=0.01$ sample (batch I) at different magnetic fields. The solid line is the best linear fit for $H=0$. The inset shows the decrease of localization length (L) with magnetic field.

field. We find that ρ vs T continues to fit approximately the VRH expression [Eq. (1)] with the same value of n for different magnetic fields up to the highest magnetic field (20 T) which is shown in Fig. 4. It is worth mentioning that we have not seen more than a 2% change in the prefactor (ρ_0) up to 20 T. The slope of $\ln\rho$ vs T^{-n} increases very slowly with magnetic field and becomes insensitive at high fields. This increase of T_0 with field indicates a decrease of the localization length [Eq. (2)] provided the density of states, $N(E)$, is field independent. Assuming B in the expression for the density of states to be constant, we have estimated the decrease of the localization length $L(H)$ using Eq. (2) with $k=1$. The variation of the localization length $[L(H)/L(0)]$ with magnetic field is shown in the inset to Fig. 4. From the figure it is clear that $L(H)$ decreases very slowly with increasing field and then saturates at high fields. Even though the resistivity increases by more than a factor of 2 at 20 T, the localization length decreases by 5% only. The effect of a magnetic field on the localization length is much weaker in CB polymers in comparison to that of doped semiconductors[3,24] However, the variation of L with H in CB polymers is very similar to Pr-doped $YBa_2Cu_3O_7$ systems where a small decrease in L is observed.[20]

IV. CONCLUSION

In conclusion, we have observed that the temperature dependence of the resistivity of CB polymers follows the variable-range-hopping expression with a large value of the exponent n and a small value of T_0 which is independent of the concentration. This large value of n and the constant T_0 value are explained assuming superlocalization of electronic states in the percolation network of the carbon-black polymer. We observed that the magnetoresistance is small and negative at high temperatures while it is large and positive at low temperatures. Like T_0 and n, the magnetoresistances of the different samples are also concentration independent.

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