Anomalous magnetoresistance of carbon-doped EuB_6: Possible role of nonferromagnetic regions

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We report on electrical, magnetic, and thermal properties of $EuB_{5.99}C_{0.01}$. The observed anomalously large negative magnetoresistance is attributed to the effect of fluctuations in carbon concentration. In accordance with our interpretation, in the bulk ferromagnetic (FM) state the carbon-rich regions give rise to helimagnetic domains, which are responsible for an additional scattering term in the electrical resistivity. Above the temperature of the bulk FM ordering, T_C=4.3 K, we suppose that the carbon-rich regions act as "spacers" preventing magnetic polarons (MPs) to link, to form FM clusters, and eventually to percolate. As we propose, such spacers, being in fact volumes incompatible with the existence of MPs (and FM state in general), may be responsible for a decrease in the percolation temperature and for an additional (magneto)resistivity increase in systems with MPs and other spatially inhomogeneous materials containing FM phase.

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EuB_6 is a rare example of low carrier density hexaboride that orders ferromagnetically at low temperatures and undergoes a metal-insulator phase transition. The ferromagnetic (FM) order is established via two consecutive phase transitions;[1–3] the specific heat exhibits a broad maximum suggesting a magnetic transition at T_C=12.6 K and an additional small λ-like anomaly with a maximum at 15.5 K.[3] Also the differential resistivity, $d\rho/dT$, indicates two consecutive transitions.[3,4] The high-temperature magnetoresistance is large and negative, increasing in absolute value with decreasing temperature up to almost 100% around 15.5 K.[4] The positive magnetoresistance in FM regime reaches a value of up to 700% at 7 T and 1.7 K.[4]

Physical properties of EuB_6 are thought to be governed by magnetic polarons (MPs).[3,5–7] Raman-scattering measurements[5] indicate appearance of MPs in EuB_6 during the cooling at about 30 K. As suggested by Süllow *et al.*,[3] the magnetic phase transition at 15.5 K represents the emergence of spontaneous magnetization accompanied by metalization. At this temperature MPs begin to overlap and form a conducting, ferromagnetically ordered phase that acts as a percolating, low-resistance path across the otherwise poorly conducting sample.[3] With decreasing temperature the volume fraction of the conducting FM phase expands until the sample becomes a homogeneous conducting bulk ferromagnet at 12.6 K.[3] Paschen *et al.*[4] and Yu *et al.*[7] regard 15.5 K as the FM ordering temperature, T_C.

Because of the very low number of intrinsic charge carriers ($\sim 10^{20}$ cm^{-3}),[8] even a slight change in the concentration of conduction electrons, e.g., due to a change in chemical composition or number of impurities, can drastically modify the electric and magnetic properties of EuB_6.[9,10] A way to bring electrons into the conduction band of EuB_6 is to substitute boron by carbon since carbon is of similar size and has one more electron in comparison to boron. It seems that the prevailing effect of electrons added to the system is to produce antiferromagnetic exchange which competes with FM interactions existing in EuB_6.[11] As shown by neutron-diffraction studies,[11] the predominant FM ordering in the stoichiometric EuB_6 changes with increasing carbon content through a mixture of FM and helimagnetic (HM) domains into purely antiferromagnetic (AFM) state.[11] The HM domains are associated with carbon-rich regions characterized by higher carrier density than the surrounding matrix.[11] A different impact of the RKKY interaction due to distinct carrier density yields to the different type of magnetic order.[11] The paramagnetic Curie temperature of $EuB_{6-x}C_x$ changes its sign for x=0.125.[9]

In this paper we report on $EuB_{6-x}C_x$, with $x \approx 0.01$. We already reported more than a decade ago the unusual transport properties of the single crystal from the same batch.[12] The observed behavior is quite different than that of the stoichiometric EuB_6. The residual resistivity is exceptionally high, even higher than the room-temperature resistivity. Application of magnetic field of 3 T at 4.2 K causes a dramatic reduction in the resistivity yielding $\rho(0\ T)/\rho(3\ T)=3.7$. We have ascribed the huge residual resistivity to the scattering of conduction electrons on a mixed magnetic structure.[12] Now we present new results of electric, magnetic, and heat-capacity measurements and discuss the magnetotransport properties in comparison to the stoichiometric EuB_6. The obtained data support the aforementioned hypothesis that the dominant scattering process at temperatures below the bulk magnetic transition originates in the mixed magnetic structure. Taking into account the evidence of MPs in a similar system, $Eu_{1-y}La_yB_6$ with $y \leq 0.01$,[5] we interpret the observed behavior supposing the presence of MPs. We argue that regions of proper size and space distributions incompatible with MPs (or with FM ordering in general), so-called "spacers," represented in the case of $EuB_{5.99}C_{0.01}$ by carbon-rich regions, can be the clue for understanding the origin of the colossal negative magnetoresistance in spatially inhomogeneous systems containing MPs (or conductive FM phase in general).

All samples studied in this work were cut from the same single crystal grown by means of the zone floating used in

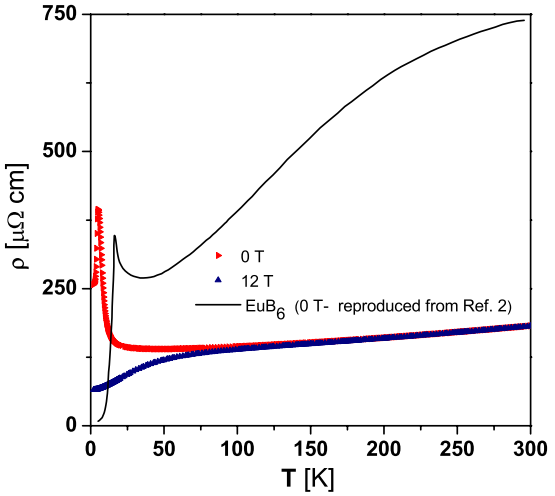

FIG. 1. (Color online) Temperature dependence of the electrical resistivity of $EuB_{5.99}C_{0.01}$. For comparison, digitalized data for EuB_6 published by Süllow *et al.* (Ref. 2) are depicted.

our previous studies.[12] Recent microprobe analysis of the crystal has revealed carbon content corresponding to the stoichiometric formula $EuB_{5.99}C_{0.01}$. The electrical resistance, magnetoresistance, heat capacity, and ac susceptibility were measured in Quantum Design PPMS and MPMS. A correlation of the $\rho(T)$ dependences with those published previously[12] is an indication of a good time stability of the electric properties. The direction of the applied magnetic field was perpendicular to electrical current in all magnetoresistance measurements.

As shown in Fig. 1, the electrical resistivity of $EuB_{5.99}C_{0.01}$ slightly decreases upon cooling from 300 K until it reaches a shallow minimum at about 40 K. Below 10 K the $\rho(T)$ increases steeply, passes a maximum at $T_{RM} \approx 5$ K, and subsequently falls off, having the tendency to saturate at lowest temperatures.[12] Magnetic field of 12 T suppresses the maximum totally. To show the huge difference from the stoichiometric EuB_6, a digitalized $\rho(T)$ dependence published by Süllow *et al.*[2] is also depicted in the figure.

The low-temperature part of $\rho(T)$ dependences at distinct magnetic fields is shown in Fig. 2. The temperature derivative of the behavior corresponding to the zero magnetic field, depicted in the left figure inset, shows a sharp maximum at T_m=4.1 K, indicating a proximity of magnetic phase transition. Since the optical reflectivity data of the studied system have not revealed any change in the plasma frequency between 4.2 and 300 K and in the temperature range of 4.2–20 K there is even no observed effect of the temperature on the infrared spectra in the whole investigated frequency region,[12] the charge-carrier concentration can be regarded as temperature independent. Taking into account that electron-phonon scattering in the isostructural LaB_6 can be neglected below 20 K,[13] we analyzed the $\rho(T)$ dependence in the paramagnetic region below 20 K using the simplified formula used at $\rho(T)$ analysis of $Eu_{1-x}Ca_xB_6$,[14] $\rho(T)=\rho_0+A.T/(T-\theta_P)$, where ρ_0 does not depend on temperature and the second term is due to spin disorder; the fitting parameter θ_P is the paramagnetic temperature. The right figure inset illustrates that such a model, with the fitting parameter θ_P=7.16 K,

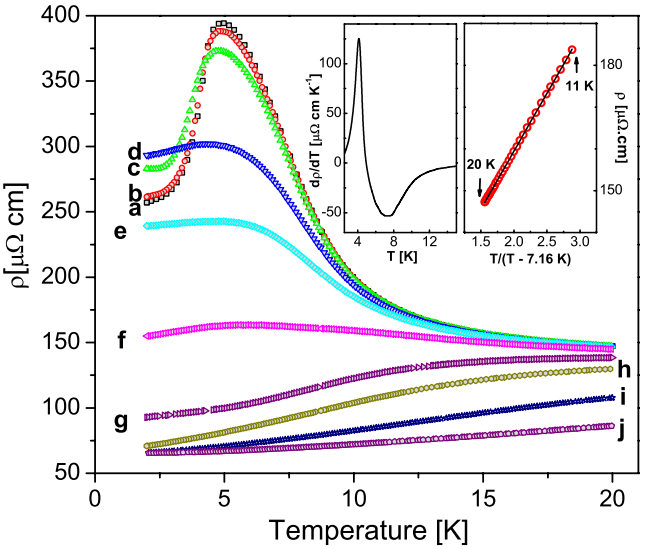

FIG. 2. (Color online) Temperature dependences of the resistivity of $EuB_{5.99}C_{0.01}$ in magnetic fields (a) 0 T, (b) 50 mT, (c) 0.1 T, (d) 0.3 T, (e) 0.5 T, (f) 1 T, (g) 2 T, (h) 3 T, (i) 6 T, and (j) 12 T. The insets show the $d\rho/dT$ vs T (left) and $\rho(T)$ vs $T/(T-7.16$ K) (right) dependences for the zero magnetic-field data.

provides an excellent description of the data between 11 and 20 K.

Temperature dependences of the magnetoresistance $MR=[\rho(B)-\rho(0)]/\rho(0)$ for selected values of the applied magnetic field between 50 mT and 12 T, derived from the data shown in Fig. 2, are shown in Fig. 3. The absolute value of MR reaches a maximum of about 0.83 for 12 T coinciding with the maximum in $\rho(T)$. Upon further cooling MR decreases continuously in absolute value. However, unlike in EuB_6,[4] MR passes through zero and reaches positive values only in the smallest magnetic fields up to 0.3 T and at lowest temperatures. This principal difference from EuB_6 is associated with the mixed magnetic structure, consisting of the FM matrix and the HM domains associated with carbon-rich regions. Sufficiently high magnetic field makes the HM do-

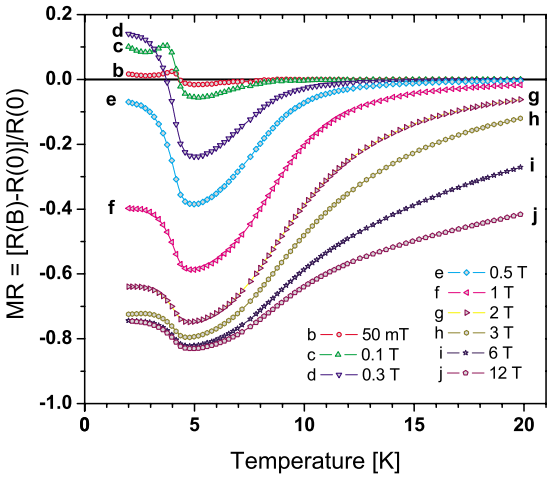

FIG. 3. (Color online) Temperature dependences of the magnetoresistance $MR=[\rho(B)-\rho(0)]/\rho(0)$ derived from the data in Fig. 2. The solid lines are a guide to the eyes.

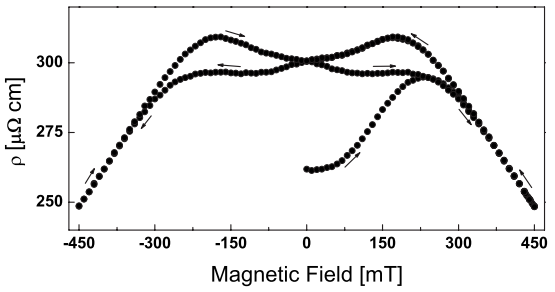

FIG. 4. Hysteresis of magnetoresistance of $EuB_{5.99}C_{0.01}$ at 2 K. After cooling from 30 K in zero magnetic field, the field was increased up to 450 mT, then decreased to −450 mT, and finally increased to 450 mT. The arrows show the emergence of the curve.

mains energetically unfavorable and therefore reduces their volumes (and probably destroys them completely at highest fields), giving rise to negative magnetoresistance.

As shown in Fig. 4, a significant hysteresis of the resistivity at 2 K appears for $|B| \leq 0.3$ T. Also a not negligible hysteresis of magnetization was observed in the same interval of magnetic field.[15] This suggests that the positive magnetoresistance in the lowest magnetic fields may be due to the conduction-electron scattering on the domain walls within the FM matrix.

The magnetoresistance measured between 2 and 20 K, depicted in Fig. 5, reveals two temperature intervals with different magnetoresistance regimes: (i) below 4.2 K—the resistivity is enhanced by small magnetic fields ($B \leq 0.3$ T) and reduced by higher fields ($B \geq 0.5$ T); (ii) above 4.7 K—the resistivity monotonically decreases with increasing applied magnetic field. A *qualitative* change in magnetoresistance regime in the temperature region between 4.2 and 4.7 K might be a sign for a magnetic phase transition in this temperature interval.

With the aim to get more information on the magnetic

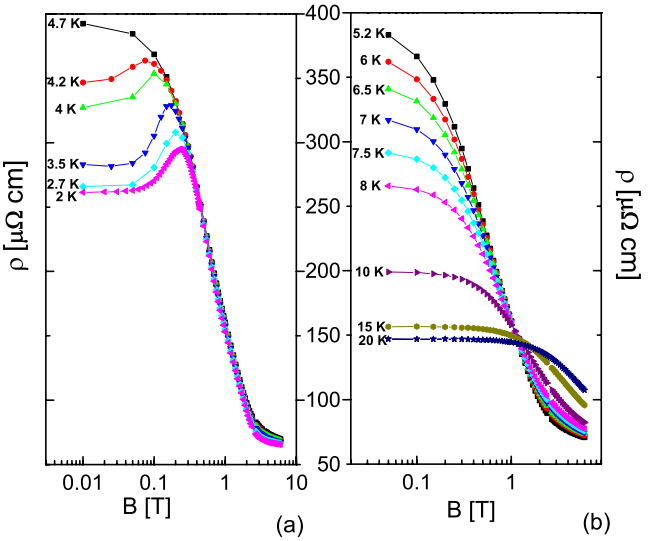

FIG. 5. (Color online) Resistivity dependence on the magnetic field measured at selected temperatures (a) below the temperature of the resistivity maximum and (b) above this temperature. The solid lines are a guide to the eyes.

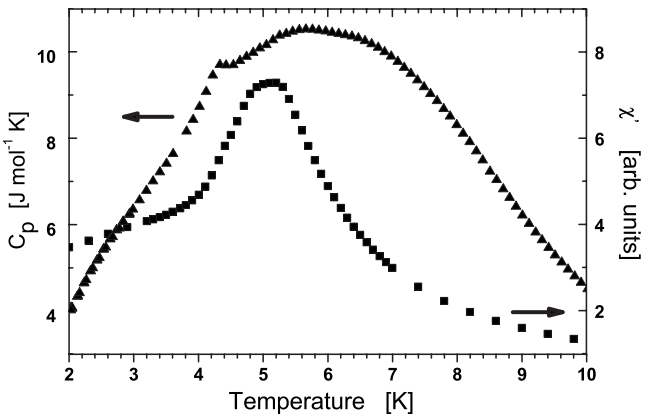

FIG. 6. Temperature dependences of the real part of the ac susceptibility, $\chi'(T)$, and the specific heat, $C_p(T)$.

properties and the phase transition(s), the specific heat was measured in the temperature range of 2–30 K. The $C_p(T)$ dependence below 10 K, depicted in Fig. 6, is different from that of EuB_6 (Ref. 3) but similar to that reported earlier for $EuB_{5.97}C_{0.03}$.[16] A small λ-like anomaly appears at 4.3 ± 0.1 K, i.e., near above the temperature of the sharp peak in $d\rho/dT$, in the aforementioned interval in which the presence of magnetic phase transition is indicated by the change in the magnetoresistance behavior. We regard this as a sign of the bulk FM phase transition at $T_C = 4.3$ K. At higher temperatures a broad hump with a maximum at 5.7 K is observed. This is a difference from the stoichiometric high quality EuB_6, where a small λ-like anomaly, coinciding with one in $d\rho/dT$, is observed *above* the bulk FM transition, reflecting itself in the broad peak in $C(T)$ with the maximum at $T_C = 12.6$ K.[3]

The real part of the magnetic susceptibility, $\chi'(T)$, measured in the temperature range of 2–300 K, follows the Curie-Weiss behavior at high temperatures. Fitting the χ' between 30 and 300 K to the Curie-Weiss function yields the paramagnetic temperature, $\theta_p = 7.8$ K. As can be seen from Fig. 6, the $\chi'(T)$ dependence with a maximum coinciding with the maximum in $\rho(T)$ at $T_{RM} \approx 5$ K, changes its slope in proximity of T_C. The other inflection point in the $\chi'(T)$ dependence correlates with the position of the broad peak in $C_p(T)$.

Temperature and field dependences of the magnetization were measured too. The data have been replotted as M^2 vs H/M (see Fig. 7). However, it has been found that an extrapolation of the curves from high-field, large M data according to the formula $H/M = a_0 + a_1 M^2 + A_2 M^4 + \ldots$ does not yield a consistent result. (Except for the lowest temperatures, where a linear fit yields a well-defined intercept point with the M^2 axis, it is not possible to fit the data according to this formula.)

Vanishing of the spontaneous magnetic moment was measured by examination of induced-(remanent)-magnetization quenching at increasing temperature. The first attempts in a commercial SQUID magnetometer showed that the measurement is strongly affected by trapped magnetic field in the superconducting magnet during an initial magnetization of the sample. To avoid these effects we decided to measure a

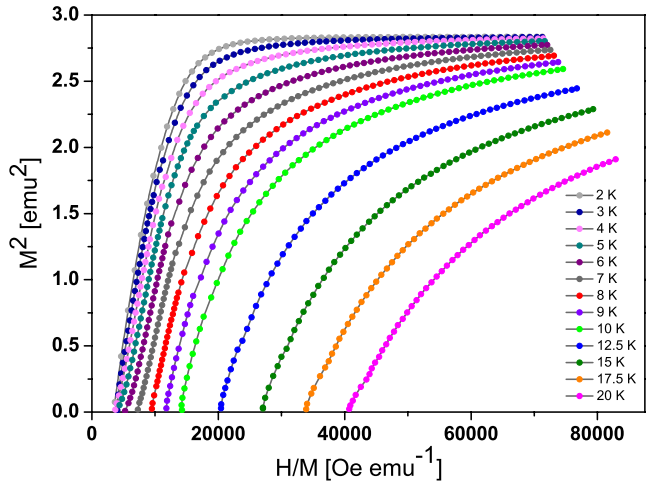

FIG. 7. (Color online) Arrot plots.

change in remanent magnetization in ultra-low-field noncommercial SQUID magnetometer (with residual magnetic field lower than 1 μT) operating above the temperature of liquid helium. An experimental procedure was as follows. First, the sample was cooled from a temperature above 30 K close to the liquid-helium temperature in *zero* magnetic field. Consequently the sample was magnetized in magnetic field of 2 mT, and finally, after slow removing of the magnetic field, the sample was slowly warmed up to 20 K at simultaneous recording of the (remanent) magnetic moment in the temperature interval of 4.5–20 K. As can be seen in Fig. 8, the magnetic moment persists up to ~9 K, which reveals the presence of ferromagnetic phase above the T_C=4.3 K. A marked change in the slope followed by a steep increase is observed at ~4.8 K, coinciding well with the temperature of the resistivity maximum. The intermediate region between 4.8 and 9 K coincides with the broad maximum in the specific heat.

As shown above, the obtained data reveal essential differences between the studied material and the stoichiometric EuB_6. On the other hand there are remarkable similarities

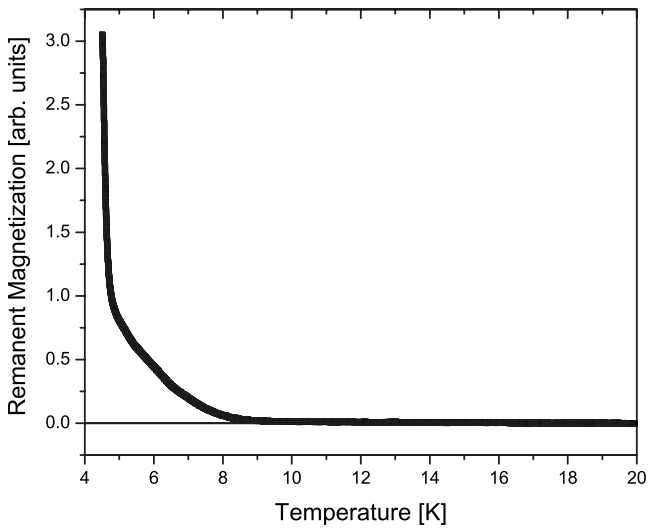

FIG. 8. Temperature dependence of remanent magnetization.

with carbon-doped system $EuB_{1-x}C_x$ in the "intermediate" state corresponding to x=0.03–0.05.[10] As already mentioned, the magnetic order of carbon-doped EuB_6 evolves from FM in near-stoichiometric material to an incommensurate spiral structure in $EuB_{5.8}C_{0.2}$, passing through an intermediate state with inhomogeneous exchange.[11] In accordance with the neutron-diffraction data, the intermediate state could be interpreted either in terms of a mixture of FM and HM domains or as a single phase with a conical spiral structure.[11] Since the available anisotropy is not strong enough for Eu^{2+} in a simple cubic lattice to stabilize a conical spiral structure, the former interpretation is favored.[11] The neutron-scattering diagram recorded in magnetically ordered state moreover shows a broad additional scattering under each nuclear peak that could be associated with small incoherent regions, magnetically ordered, whose size would be about 5 nm.[11] These regions could be associated with local fluctuations in carbon concentration, producing carbon-rich HM domains and carbon-poorer regions where the balance of exchange interaction with neighboring Eu atoms is below the threshold required for the appearance of helimagnetism.[11] With respect to the neutron-diffraction data it has been concluded that the magnetic structure of $EuB_{5.95}C_{0.05}$ can be described as a mixture of FM phase and HM domains.[11,17] In accordance with our investigations, showing similar transport properties of the studied system as those of $EuB_{5.97}C_{0.03}$ and $EuB_{5.95}C_{0.05}$,[10] we reasonably suppose that $EuB_{5.99}C_{0.01}$ belongs to the intermediate magnetic region in the phase diagram of carbon-doped EuB_6 so that the ground state of the studied system can be considered to consist of FM matrix and HM domains, whereas the latter originate in carbon-rich regions. According to our opinion, the inhomogeneous distribution of carbon, yielding the carbon-rich regions, is the clue for understanding this compound. As discussed below, the observed magnetotransport properties can be consistently explained, taking into account a specific role of these regions.

An important issue that should be taken into consideration is an eventual presence of MPs in the studied material. The relevance of MPs might be indicated, e.g., by consistent analysis of electrical resistivity and magnetization.[3] However, in contrast to EuB_6, where such an analysis was successfully performed,[3] the high-field resistivity of the studied compound monotonically decreases in the temperature interval of 2–8 K even at magnetic field of 14 T, as shown in Fig. 9. This makes it impossible to isolate the negative part of the magnetoresistance by fitting and subtracting the high-field magnetoresistance, $\rho_{\mathrm{met}} \propto H^2$, from the measured $\rho(H,T)$.[3] Another sign of presence of MPs is an exponential behavior of $\rho(T)$, typical for the magnetic polaron hopping conduction. The fact that such a behavior was not found in the studied system is considered to be a consequence of the presence of carbon-rich regions (with higher concentration of conduction electrons and with electrical conduction superior to that originating from the hopping process) representing weakly conducting electrical interconnections between the MPs.

In spite of this, since the presence of MPs was confirmed in $Eu_{1-y}La_yB_6$ with $y \leq 0.01$,[5] taking into account similarity of this system with the studied carbon-doped one, (the sub-

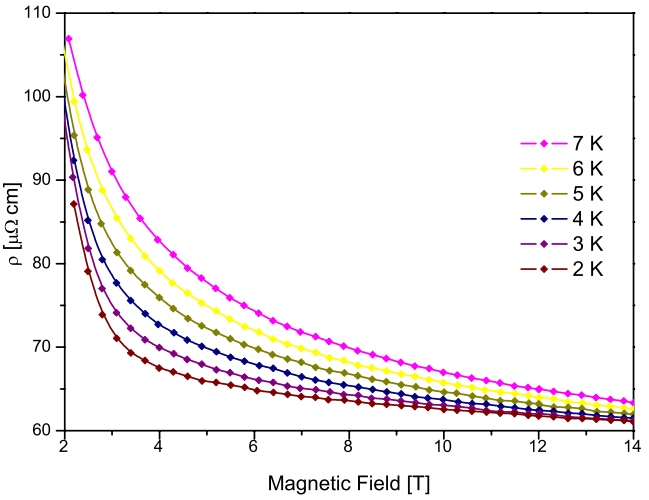

FIG. 9. (Color online) Magnetic-field dependences of the resistivity in the temperature range of 2–7 K up to 14 T.

stitution of Eu by La introduces one electron per La atom analogously like the substitution of boron by carbon; the qualitative phase diagrams have lot of common features—the magnetically ordered state is FM at lowest levels of La/C-substitution and becomes mixed, consisting of FM and AFM/HM phase for higher La/C concentrations) it is reasonable to suppose that MPs are present in $EuB_{5.99}C_{0.01}$, too. Moreover, the above presented measurement of remanent magnetization have indicated the presence of FM phase up to ~9 K, which is an observation qualitatively consistent with MP scenario.

Taking into account the discussion above we suppose that MPs form above the T_C in $EuB_{5.99}C_{0.01}$. However, since MPs can exist only in a low carrier density environment, we suggest that, with the difference from EuB_6, the presence of MPs is not allowed in the whole volume. We suppose that due to too high electron density, the regions with carbon content exceeding a certain limit are not compatible with the existence of MPs (and with FM state in general). So, MPs form only outside the carbon-rich regions. As the temperature decreases MPs increase in volume and eventually become *linked*,[22] forming FM clusters, namely, aggregates of polarons into a single FM region.[18] On the other hand, carbon-rich regions order helimagnetically at sufficiently low temperature. Moreover, the fluctuation of carbon content causes the characteristic temperature of the magnetic phase transition (HM and FM) to spread out into a temperature interval of a finite width. This, as we suppose, reflects itself in the broad peak of $C_p(T)$ with the maximum at 5.7 K. The observed gradual appearance of magnetic moment below 9 K (see Fig. 8), coinciding with the broad maximum in the specific heat, supports such an interpretation. Upon further cooling the FM clusters grow in size until the bulk ferromagnetism occurs at the temperature of the small specific-heat anomaly, T_C=4.3 K.

According to our proposition the essential impact of the carbon-rich regions on the magnetotransport properties of the investigated compound can be associated with the fact that carbon-rich regions prevent linking and percolation processes of MPs. Assuming an analogous scenario as that of Süllow *et al.*,[3] mentioned in the introductory part of this paper, the carbon-rich regions should act as some spacers prohibiting the formation of a conducting, ferromagnetically ordered path across the $EuB_{5.99}C_{0.01}$ sample. As a consequence, the system persists in a poorly conducting state down to lower temperatures. Due to the extension of the temperature interval, in which the resistivity increases upon cooling, an additional resistivity increase is observed, resulting in the higher value of the resistivity maximum. Such a scenario explains why the value of the resistivity maximum observed for $EuB_{5.99}C_{0.01}$ (~390 $\mu\Omega$ cm at ~5 K) is larger than that of the stoichiometric EuB_6 (~350 $\mu\Omega$ cm at ~15 K),[2] although $EuB_{5.99}C_{0.01}$ is at room temperature about four times better conductor, having $\rho(300\ \mathrm{K}) \approx 180\ \mu\Omega$ cm, than EuB_6 with $\rho(300\ \mathrm{K}) \approx 730\ \mu\Omega\ \mathrm{cm}^2$ (see Fig. 1).

In accordance with the discussion above we summarize principal differences between the pure EuB_6 and the investigated $EuB_{5.99}C_{0.01}$ as follows. (i) While the paramagnetic state in EuB_6 is homogeneous, the paramagnetic state in $EuB_{5.99}C_{0.01}$ has to be treated as inhomogeneous, containing regions with increased carbon content and with correspondingly *higher* electrical conductance in comparison to the remaining matrix. (ii) The magnetic polaron phase in EuB_6 can be treated as a two-component system, consisting of highly conducting FM phase represented by MPs and poorly conducting paramagnetic matrix. This phase in $EuB_{5.99}C_{0.01}$ does have three components at least: highly conducting FM phase represented by MPs, regions with lower carbon content, and carbon-rich domains. The latter, incompatible with the existence of MPs due to too high charge carrier concentrations, work as spacers, preventing MPs to link and to form a conductive path across the sample. (iii) Finally, in the magnetically ordered state EuB_6 is a homogeneous ferromagnet, while $EuB_{5.99}C_{0.01}$ can be treated as two-component system consisting of FM matrix and HM domains formed in the carbon-rich regions.

The above explanations are speculative, and further research—e.g., detailed neutron-diffraction studies—could greatly elucidate the real microscopic structure of the studied sample and the relevance of MPs. Nevertheless, the proposed scenario allows to explain (at least partially) the anomalous magnetotransport of the studied carbon-doped EuB_6 and to understand the differences with the stoichiometric EuB_6. After all, it should be noted that MPs are not necessary to describe the behavior of the studied system. It is also possible to consider a continuous ferromagnetic transition with gradual increase in the volume of ferromagnetic phase that forms first in regions with less carbon content. Until the bulk ferromagnetism occurs, the regions with increased carbon content will work as spacers. Notwithstanding, we consider the latter case to be less probable, such as the existence of MPs being well established in materials similar to the studied system.

An important impact of the supposed role of spacers is that such entities possibly should play a crucial role also in other spatially inhomogeneous systems with MPs or with FM phase in general. However, it should be mentioned that spacers, which are electrically *non*conductive, or have *lower* electrical conductance than the paramagnetic surroundings

should be even more effective and should be responsible for even higher (negative) magnetoresistance than spacers represented by carbon-rich regions having higher electrical conductance than the surrounding matrix. Indeed, studies of $Eu_{1-x}Ca_xB_6$ have revealed colossal magnetoresistance[4,14,19] that is several orders of magnitude greater than that of the investigated carbon-doped EuB_6. According to our opinion this can be a consequence of low-conductive spacers represented by calcium-rich regions. A natural consequence of the proposed scenario is that it should show a route for effective increasing in magnetoresistance by introducing non-FM spacers into the system with FM phase. We would also like to remark that similar concepts based on phase separation have been discussed also in manganites[23] and that the performed calculations[20] are in good qualitative agreement with the proposed role of the spacers. With respect to a possibility to tune the properties of magnetoresistive materials, we would like to point out an interesting analogy between the proposed scenario of the magnetoresistance enhancement and the role of (nonsuperconducting) pinning centers in increasing the critical magnetic field in superconductors.

In summary, our studies indicate that the anomalous transport properties of $EuB_{5.99}C_{0.01}$ can be consistently explained, considering the noncompatibility of carbon-rich regions with the existence of MPs and FM phase. The broad peak in the heat capacity with the maximum at 5.7 K is associated with the formation of FM clusters in carbon poor regions. The small anomaly in $C_p(T)$ at T_C=4.3 K is ascribed to the percolation of FM clusters and to the bulk FM transition. The peak in the $d\rho/dT$ vs T dependence at T_m, lying slightly below T_C, is considered as a sign of rapid enhancement of the conductivity in the bulk FM phase. The unusually high value of the electrical resistivity maximum is linked with the spacing role of the non-FM (carbon-rich) regions. Finally, we emphasize that introducing regions preventing the percolation of FM clusters might strongly enhance the magnetoresistance of systems with MPs and other spatially inhomogeneous materials containing FM phase. This might show a route for future research efforts in relation to colossal magnetoresistance.

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