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# Superconductivity and Physical Properties in the $K_xMoO_{2-δ}$

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Additional information is available at the end of the chapter

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## 1. Introduction

In a normal metallic conductor the electrical resistivity decreases until it reaches a lower limit ( $\approx 0$ ) when heat energy is removed from the system. In these materials electrons are scattered by the lattice and obey Fermi-Dirac statistics [1]. The behaviour of a gas of identical particles at low temperature depends on the spin of the particle. Fermions are formed by half-integral spin, and obey Pauli's exclusion principle, wherein two of them cannot have the same quantum numbers [1]. However many metals, alloys and compounds exhibit the electrical resistivity dropping suddenly to zero and exclude magnetic flux completely when cooled down to a sufficiently low temperature [1]. This phenomenon is known as superconductivity. It was observed first by Kamerlingh Onnes in 1911, a couple of years after the first liquefied helium [2]. At a critical temperature  $T_C$ , the material undergoes a phase transition from a normal state to a superconducting state. At that point an electron-electron attraction arises mediated by phonons and the pairs formation of electron is favourable. Electron pairs are named Cooper pairs [3,4]. Cooper pairs are a weakly bound pair of electrons, each having equal but opposite spin and angular momentum.

It is interesting to note that when a superconductor is cooled below its critical temperature its electronic properties change appreciably, but its crystalline structure remains the same, as revealed by X-ray or neutron diffraction studies. Furthermore, the formation mechanism of photons that depends on the thermal vibrations of the atoms remains the same in the superconducting phase as in the normal state. This means that superconductivity is not associated with any remarkable change in the crystalline structure of the specimens. However, although superconductivity is not a property of particular atoms, it does depend strongly on their arrangement. The conduction electrons themselves must be responsible for the superconduct-

ing behaviour. A feature that illustrates an important characteristic of these superconducting electrons is that the transition from the normal to the superconducting state is very sharp [3,4]. This could only happen if the electrons in a superconductor become condensed into a coherent, ordered state, which extends over long distances compared to the distances between the atoms. A superconducting state is more ordered than the normal state in a metal. This means that it has a lower entropy, the parameter that measures the amount of disorder in a system. In an analogous way, the entropy of a solid is lower than that of a liquid at the same temperature; solids are more ordered than liquids. So, when a material turns to a superconducting state, the superconducting electrons must be condensed into an ordered state compared to normal electrons.

But what happens if, in a material, the electronic interaction leads to the formation of Cooper pairs but in a disordered state? If this were so then any local variations from collective action between the electrons would broaden the transition over a much wider temperature range or transition could not be noticed. Thus, despite the existence of the Cooper pairs, the state formed would be unconventional metallic but not superconductor. Due to the existence of bosonic particles in the crossover from weak coupling Bardeen-Cooper-Schrieffer (BCS) pairing to a Bose-Einstein condensate (BEC) of tightly bound pairs, this unconventional metallic phase is conveniently called Bose metal [5,6]. The Bose metal has been characterized as a system without long-range order. This new state of the matter has attracted a lot of attention over the past decades due to occurrence of a metal-insulator transition in low dimensional systems [5,6]. Experiments have shown that this transition can be induced by disorder or by applying external magnetic field. When a sufficiently high magnetic field is applied in the material the Cooper pairs are completely localized turning its electrical conductivity to zero and leading to an insulating state. Within this framework a scale law was proposed to identify a Bose metal-insulator transition (BMIT). So, this transition can be analysed in terms of two-parameter scaling, as follows:

$$RT^{1+2/z} / \delta^{2\beta} = f\left(\delta T^{\frac{1}{zV}}\right), \quad (1)$$

where  $R$  is the electrical resistance and  $T$  is the temperature.  $\beta = V(z + 2)/2$ ,  $z$  and  $V$  are critical exponents, and  $\delta = H - H_c$  the deviation of the variable parameter (magnetic field) from the critical values [5].  $H_c$  is the critical magnetic field where the BMIT occurs. Such a transition has been recently observed in semimetals as graphite and bismuth [7], and also in the quasi one-dimensional  $\text{Li}_{0.9}\text{Mo}_6\text{O}_{17}$  [8]. Theoretical analysis has suggested that the BMIT in graphite is associated with the transition between Bose metal and excitonic insulator state, whereas in  $\text{Li}_{0.9}\text{Mo}_6\text{O}_{17}$  this transition is discussed in terms of localized bosons in a low dimensional system, which could be related to the Luttinger liquid (LL) state.

Another interesting subject to consider is that superconductivity and magnetism are two different ordered states into which substances can condense at low temperatures, and in general these states are opposite to each other [1]. It has generally been believed that the

conduction of electrons in a metal cannot be both ferromagnetically ordered and superconducting. In conventional superconductors, local magnetic moments break up the spin singlet Cooper pairs, and hence strongly suppress superconductivity, an effect known as pair-breaking [3,4]. However, since the 1970s several studies on the interplay between superconductivity and magnetism have arisen in the scientific community. Namely, a coexistence between ferromagnetism and Bose metal state has been demonstrated for both highly oriented pyrolytic graphite and graphite sulphur composites [9]. Actually, the authors' assumed the observation of metal-insulator crossover in both cases might be related to a Bose metal-insulator transition [9].

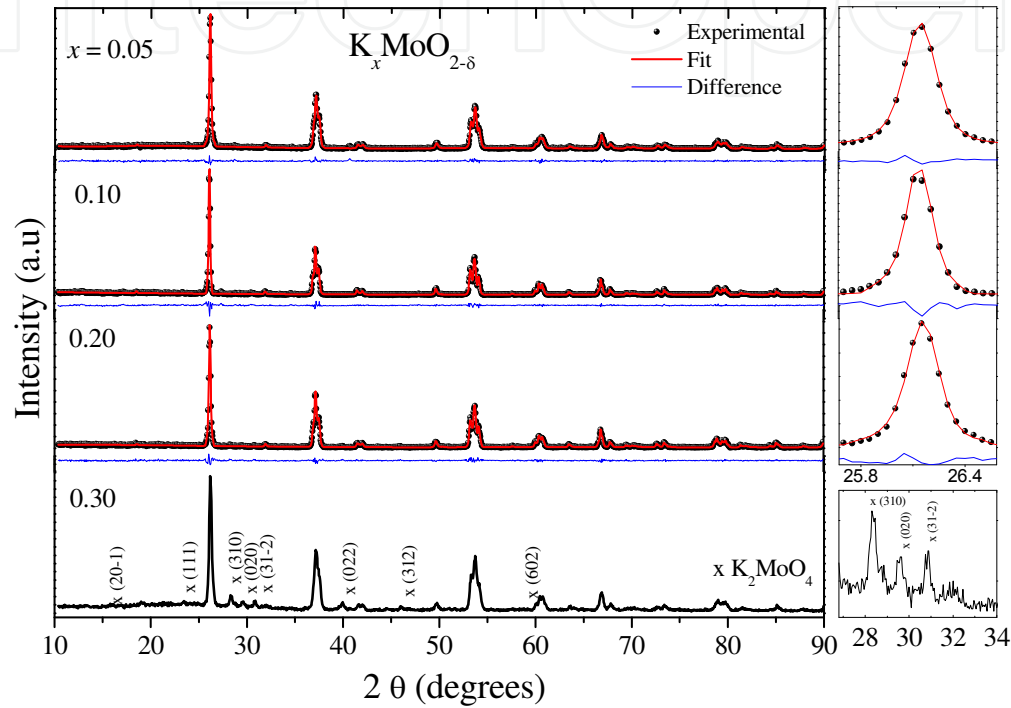
This chapter will report results about recent studies on the  $K_x\text{MoO}_{2.8}$  system, which has suggested that K doping in the  $\text{MoO}_2$  matrix causes defects leading to a magnetic ordering temperature and superconducting critical temperature ( $T_C$ ) ratio ranging from 7 to 18 in this compound [10]. These are the highest ratios reported so far for a magnetic superconductor. Furthermore, anomalous metallic behaviour has been reported in this compound [11]. The study of the anomalies provides important conclusions, such as the temperature dependence of the electrical resistance is described by a power law of the type  $R(T) = R_0 + CT^\alpha$ , where  $\alpha$  is found to be near 0.5. This value of  $\alpha$  can be interpreted according to the theory of LL. Anderson and Ogata [12] proposed a theoretical description based on the model of the Luttinger liquid (LL) in which the electrical resistivity of a one-dimensional conductor must be described by a power law of the type  $\rho(T) = \rho_0 + CT^\alpha$  where the residual resistivity is  $\rho_0$ ,  $C$  is a constant and not universal  $\alpha$  is provided in the anomalous exponent model [12,13]. This result suggests a mechanism for one-dimensional metallic conduction in the  $K_x\text{MoO}_{2.8}$  system. Furthermore, the LL theory suggests that the interaction between the conduction electrons is attractive to positive  $\alpha$  and smaller than one. Measurements of several samples with different amounts of potassium confirm the value of  $\alpha$  near 0.5 for all of them. With the goal of increasing the understanding of the physical properties of  $K_x\text{MoO}_{2.8}$  at low temperatures, especially the anomaly shown in the electrical resistivity curves below  $\sim 70\text{K}$ , magnetoresistance measurements as a function of temperature were performed. Under applied magnetic field the electrical resistance changes to larger values, indicating a positive magnetoresistance. This behaviour is very similar to a Bose metal-insulator transition observed in semi-metals such as graphite-bismuth [7], thin films of  $\text{MoGe}$  [5] and to compounds with quasi-low-dimensional behaviour such as  $\text{Li}_{0.9}\text{Mo}_6\text{O}_{17}$  [8]. This behaviour was interpreted according to the scaling theory. This theory implies the existence of Cooper pairs without long-range order, thus featuring a metallic state at absolute zero (Bose metal). This seems to agree with that observed by the LL theory and with the existence of superconductivity in the  $K_x\text{MoO}_{2.8}$  system.

### 1.1. $K_x\text{MoO}_{2.8}$ crystalline structure

Polycrystalline samples of  $K_x\text{MoO}_{2.8}$  with  $0 \leq x \leq 0.30$  were prepared in pellet forms using appropriate amounts of  $\text{Mo}$ ,  $\text{MoO}_3$ , and  $\text{K}_2\text{MoO}_4$ . The precursor  $\text{K}_2\text{MoO}_4$  was obtained after heat treating  $\text{K}_2\text{CO}_3$  and  $\text{MoO}_3$  in air at  $400^\circ\text{C}$  for 24h followed by a treatment at  $700^\circ\text{C}$  for 24h. The pellets of  $K_x\text{MoO}_{2.8}$  were sintered at  $400^\circ\text{C}$  for 1 day followed by  $700^\circ\text{C}$  for 3 days in a quartz tube under vacuum. Different heat treatments resulted in different potassium ( $x$ ) and

oxygen contents due to the volatilization of atoms inside quartz tubes. X-ray diffractometry (XRD) analysis of the samples was performed using a Shimadzu diffractometer (XRD 6000) with CuK $\alpha$  radiation and Ni filter. The 2 data were collected from 10° to 90° using step of 0.058 and counting time of 1 s.

Figure 1 shows the x-ray diffraction patterns of K $_x$ MoO $_{2-\delta}$  ( $x = 0.05, 0.10, 0.20,$  and  $0.30$ ), the final Rietveld refinement, and the difference between both.



**Figure 1.** X-ray diffraction patterns for the samples of K $_x$ MoO $_{2-\delta}$  with  $x \leq 0.30$ , Rietveld refinement and the difference between both. To right magnifications show the high quality of fit as well as the peaks belonging to impurities for the K $_{0.30}$ MoO $_{2-\delta}$  sample.

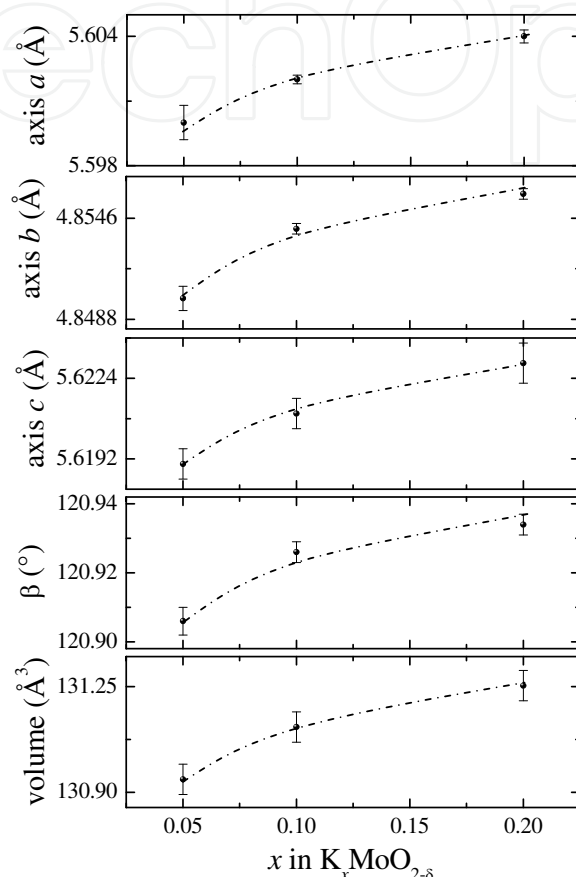
Diffractograms of all samples with  $x \leq 0.20$  reported in this work show only peaks of the MoO $_2$  phase related to the monoclinic structure (P2 $_1$ /c). The parameters, which represent the quality of the Rietveld refinement and summarizes the crystalline data description resulting from the final Rietveld refinement, are listed in the Table 1.

$x$	$a$ (Å)	$b$ (Å)	$c$ (Å)	$\beta$ (°)	$\chi^2$	$R_{wp}$
0.05	5.600(8)	4.850(7)	5.619(6)	120.906(4)	1.419	0.1238
0.10	5.602(2)	4.854(3)	5.621(6)	120.926(3)	2.213	0.1570
0.20	5.604(3)	4.856(3)	5.623(8)	120.934(3)	1.526	0.1482

**Table 1.** Rietveld quality parameters refinement and crystal parameters to K $_x$ MoO $_{2-\delta}$  samples with  $x$  between 0.05 and 0.20.

For  $x \geq 0.3$  a second phase appears related to the precursor  $K_2MoO_4$ . Its x-ray diffractogram is shown in figure 1. The existence of impurity in this specimen means that the solubility limit was reached.

In figure 2 is shown the variation of lattice parameters as a function of the K contents ( $x$ ) in the specimens.



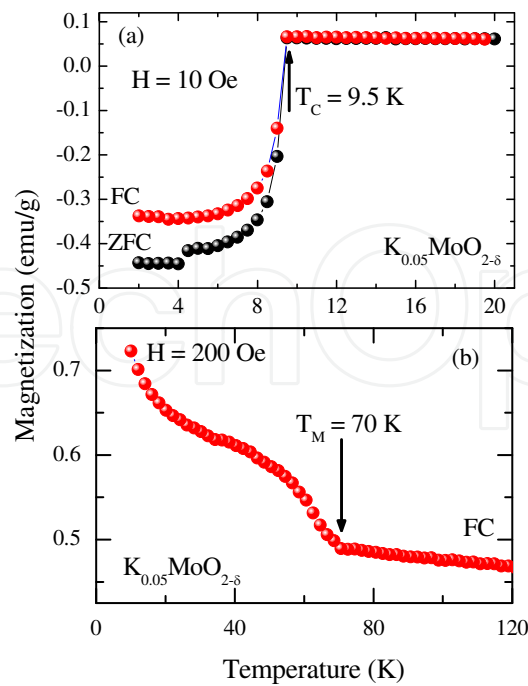
**Figure 2.** Lattice variation as a function of  $x$  in  $K_xMoO_{2.8}$ .

With increasing the amount of K one can observe a slight increase in unit cell volume, which is caused by the insertion of K in the crystalline lattice of  $MoO_2$ . For  $x = 0.05$  the unit cell volume is  $131.11(5) \text{ \AA}^3$  and for  $x = 0.20$  is  $131.25(5) \text{ \AA}^3$ , which represent an increase of approximately 1% in the volume of the unit cell.

To expand the knowledge about the physical properties of the  $K_xMoO_{2.8}$  compound was chosen for more detailed study the  $K_{0.05}MoO_{2.8}$ . So, in this part we will focus on the results for  $x = 0.05$ .

## 1.2. Interplay between superconductivity and ferromagnetism in $K_{0.05}MoO_2$

Magnetization curves as a function of the temperature measured in  $K_{0.05}MoO_{2.8}$  sample with  $H = 10$  and  $200 \text{ Oe}$  are shown in figure 3 performed under zero field cooled (ZFC) and field cooled (FC) regimes.



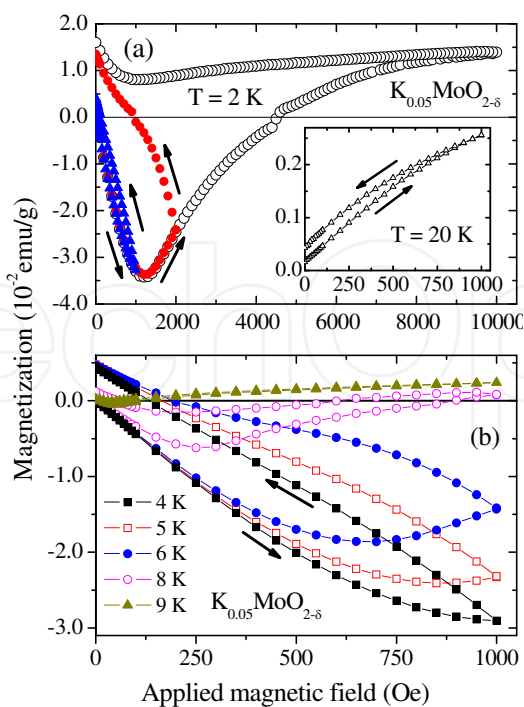
**Figure 3.** (a) ZFC and FC magnetization curves as a function of temperature for the  $K_{0.05}MoO_{2.6}$  sample under applied magnetic fields  $H = 10$  Oe. (b) FC magnetization curves as a function of temperature under  $H = 200$  Oe showing the magnetic ordering near 70K [from reference 10].

Below  $T_C \sim 9.5$  K the specimen undergoes a superconducting transition exhibiting a diamagnetic ordering. An estimation of the superconducting fraction through measurements with applied magnetic field of 10 Oe was calculated as  $\sim 0.4\%$ . The low superconducting volume fraction is probably due to the appearance of the ferromagnetic ordering at higher temperatures ( $T_M = 70$  K), which can be seen in the Figure 3 (b). The superconducting state appears below the ferromagnetic transition and the ratio between critical temperature is  $T_M / T_C \sim 7$  [10].

To study in greater detail the relationship between the ferromagnetism and superconductivity several loops of magnetization as a function of applied magnetic field was obtained above and below  $T_C$ . The results are shown in Figure 4.

Hysteresis curves were obtained with applied magnetic field ranging from 0 to 10 kOe at a temperature of 2 K, as shown in Figure 4 (a). It was observed that for values of applied magnetic field up to 1 kOe, the magnetization curve is almost linear as a function of applied magnetic field and is reversible (blue curve) due to the Meissner effect. This result allows one to estimate the lower critical field ( $H_{C1}$ ) as approximately 1.17 kOe [10]. It is quite difficult to estimate the value of the upper critical field ( $H_{C2}$ ) due to the interplay between weak ferromagnetism and superconductivity. A rough estimation, considering the measurements at 2 K, allows us to assume a value of  $H_{C2} \sim 10$  kOe, since the magnetic moment reaches saturation near to this field. With these values, it was found  $\kappa = 4.1$  to 2 K in the sample with  $x = 0.05$ , indicating that the material is a type II superconductor [10].

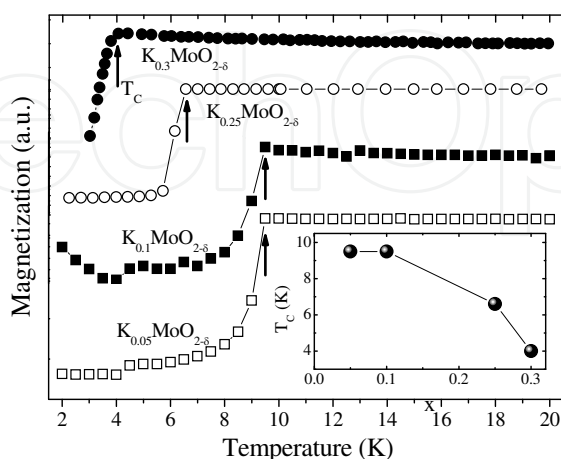
In the inset of the figure 4 (a) is shown the  $M(H)$  at 20 K. We can observe clearly a ferromagnetic ordering very similar to that observed in magnetic superconductors such as



**Figure 4.** Magnetization curves as a function of magnetic field for  $K_{0.05}\text{MoO}_{2.8}$  measured at (a) 2K using a maximum applied magnetic field of 1kOe (triangle), 2kOe (full circle), and 10kOe (open circle). (b) M versus H curves are shown at 4, 5, 6, 8, and 9K. Inset displays the M(H) curve at 20K [from reference 10].

$\text{RuSr}_2\text{GdCu}_2\text{O}_8$  [14]. In the figure 4 (b) is displayed the continuous disappearance of superconductivity when the temperature is increasing. At 9 K the superconductivity almost disappears, leaving only a weak positive magnetic component.

Figure 5 shows magnetization curves as a function of temperature for several samples with different starting compositions. The superconducting critical temperature of each sample is indicated by arrows on the figure.



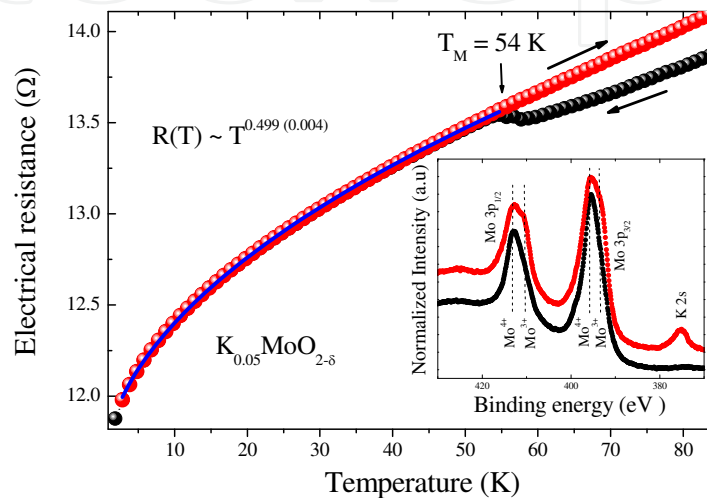
**Figure 5.** Diamagnetic transition for four samples with different starting compositions ( $x$ ). Arrows indicate the superconducting  $T_c$  for each potassium composition. Magnetization curves are shifted and shown in arbitrary units for clarity. The inset shows the superconducting  $T_c$  as a function of the starting composition for the  $K_x\text{MoO}_{2.8}$  compound [10].



The inset shows the variation in superconducting critical temperature ( $T_C$ ) as a function of initial amount of potassium in the  $K_x\text{MoO}_{2-\delta}$ . The result shows also a systematic decrease of the  $T_C$  with the increasing of the amount of K. However, the lower limit of doping with K that exhibits superconductivity is still to be determined. Recently, Parker *et al.* have reported superconductivity near 12 K for non-doped  $\text{MoO}_{2-\delta}$  [15].

### 1.3. Unconventional metallic behaviour

Figure 6 shows the electrical resistance as a function of temperature below 80 K for  $\text{K}_{0.05}\text{MoO}_{2-\delta}$ .



**Figure 6.** Electrical resistance as a function of temperature below  $\sim 80$  K for  $\text{K}_{0.05}\text{MoO}_{2-\delta}$ . The blue line is a representation of the fit by power law with anomalous exponent  $\alpha$ . The inset shows the core level photoemission spectra for the  $\text{MoO}_2$  and  $\text{K}_{0.05}\text{MoO}_{2-\delta}$  near the Mo  $3p_{1/2}$ , Mo  $3p_{3/2}$ , and K  $2s$  peaks [from reference 10].

The electrical resistance temperature dependence for  $\text{K}_{0.05}\text{MoO}_2$  sample shown in figure 6 exhibits an anomalous metallic behaviour. The blue line is the best fit using the power law such as  $R(T) = R_0 + CT^\alpha$ . The anomalous exponent ( $\alpha$ ) indicated for the electrical resistance as a function of temperature is the best value found by the fit obtained by the method of least squares. This value of  $\alpha$  suggests that the one-dimensional conduction mechanism is described by Luttinger liquid theory, with attractive interaction between electrons [8,12-13]. Furthermore, this result confirms the  $\alpha \sim 0.5$  that has also been observed for several other samples of this system [16], showing that the anomalous metallic behaviour is characteristic of this material.

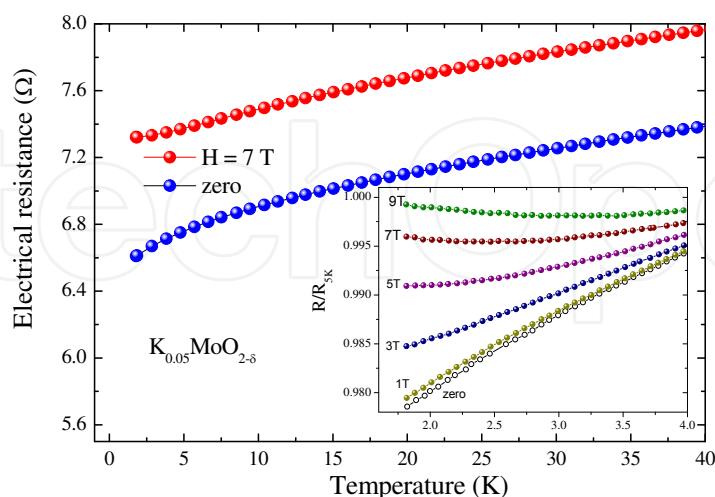
The inset of figure 6 shows the core level photoemission spectroscopy for  $\text{MoO}_2$  and  $\text{K}_{0.05}\text{MoO}_{2-\delta}$ . The Mo  $3p$  spectrum of the undoped sample shows two main structures associated with the Mo  $3p_{1/2}$  and Mo  $3p_{3/2}$  core levels, which are split due to spin-orbit effects. In the  $3p_{3/2}$  region, the peak around 395.5 eV is assigned to  $\text{Mo}^{4+}$  ( $4d^2$ ) states, but there is also a very small  $\text{Mo}^{3+}$  ( $4d^3$ ) contribution at about 393.4 eV. The latter can be associated with the covalent Mo-O bond character, which is also supported by the relatively small charge transfer satellite, at around 425 eV. Nevertheless, a small oxygen deficiency in  $\text{MoO}_2$  cannot be ruled out, as well. In the  $3p_{1/2}$  region, the corresponding binding energies of the  $\text{Mo}^{4+}$  and  $\text{Mo}^{3+}$  states are

413.0 and 410.9 eV, respectively. The spectrum of the potassium-doped sample shows a new structure at 376 eV, assigned to the K 2s core level. More importantly, the peaks due to the  $\text{Mo}^{3+}$  states in the Mo 3p core level increase and become more prominent. This relative increase indicates that the potassium ions, when incorporated into the  $\text{MoO}_2$  structure, are effectively doping the Mo 4d band with electrons, from a nominal  $4d^2$  to  $4d^3$ . The extra charge introduced in the Mo 4d band by the potassium ions would lead to an effective magnetic moment and might be the origin of magnetism in this material. A similar effect, namely, extra charge induced by the presence of ligand vacancies, was reported as the origin of ferromagnetism in other oxides with nonmagnetic ions. Similarly the electrical behaviour of the  $K_x\text{MoO}_{2.8}$  can be also influenced by this mechanism, causing the appearance of anomalous resistive electrical behaviour [10,11].

#### 1.4. Bose-metal insulator transition

With the goal of increasing the understanding of the physical properties of  $K_x\text{MoO}_{2.8}$  at low temperatures, especially the anomaly observed in the curves of electrical resistivity below  $\sim 70\text{K}$  and its relation to the existence of superconductivity, we report in this section magneto-resistance versus temperature for a sample of composition  $K_{0.05}\text{MoO}_{2.8}$ .

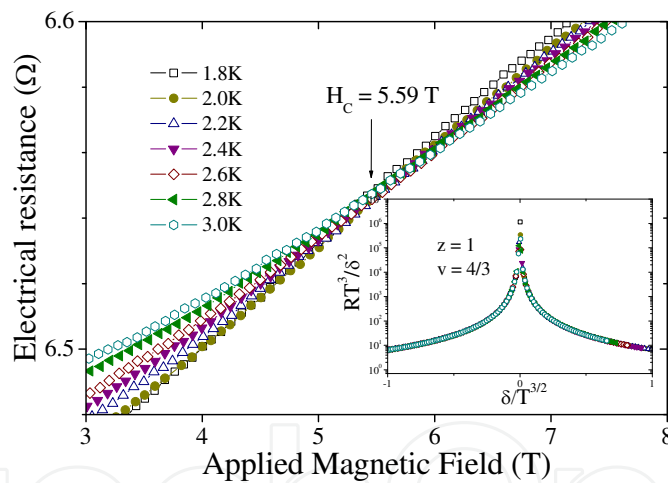
Figure 7 shows the electrical resistance as a function of temperature in a zero magnetic applied field as well as in 7 T. The results show an anomalous metallic behaviour at zero applied field. Under the applied magnetic field the electrical resistance changes to higher values indicating a positive magnetoresistance. At zero magnetic field one can observe an anomalous behaviour, in which the electrical resistance decreases as the temperature decreases. As already discussed, this behaviour differs from conventional metal-like behaviour in which the electrical resistance should reach a saturation state at low temperatures.



**Figure 7.** Electrical resistance as a function of temperature for  $K_{0.05}\text{MoO}_{2.8}$  sample under  $H = 0$  and 7 T. The inset shows the suppression of electrical anomalous behaviour below 70K. Furthermore, it displays a reduced electrical resistance  $R(T)/R(5\text{K})$  as a function of temperature for  $K_{0.05}\text{MoO}_{2.8}$  sample under several values of applied magnetic field [from reference 17].

It is important to note in figure 7 that the anomalous metallic behaviour disappears at very low temperatures ( $T < 5$  K) when magnetic field is increased. This result has attracted attention, and to obtain further information about the influence of the magnetic field on the electrical resistance behaviour at low temperatures the magnetic field dependence of the electrical resistance in the temperature range of 1.8 K to 5 K was measured. The results are shown in the inset of figure 7, which are plotted in terms of reduced temperature  $R(T)/R(5K)$  for a better comparison. At a lower applied magnetic field ( $H \leq 5$  T), electrical resistance as a function of temperature shows a metallic behaviour. At a higher applied magnetic field the electrical resistance first decreases to a minimal value, and below this temperature the electrical resistance starts to increase, leading to an insulating electrical behaviour. This behaviour is reminiscent of the transition from Bose metal to insulator (BMIT) reported for semi-metals such as graphite-bismuth [7], thin films of MoGe [5] and for compounds with quasi-one-dimensional characteristics, such as  $\text{Li}_{0.9}\text{Mo}_6\text{O}_{17}$  for example [8].

The critical magnetic field where the Bose metal-insulator transition occurs is better observed by the crossover in the isotherms shown in the Figure 8.



**Figure 8.** Isotherms showing  $H_c = 5.59$  T. In the inset we can see the Bose metal-insulator transition scaling showing the collapse near  $H_c = 5.59$  T [from reference 17].

For applied magnetic field lower than the critical value ( $H_c = 5.59$  T), the system behaves in the classical model of magnetoresistance as in metallic systems, expressed by  $\Delta R(B)/R \sim H^n$  with  $n = 2$  (typical exponent for metals) [1] for all ranges of temperature. For applied magnetic fields higher than the critical value the system deviates from this classical model with values between  $1 < n < 2$ , which features an insulating state (for detail see reference 17). The exponents obtained ( $n = 1$  and  $n = 2$ ) are similar to the values reported for graphite and bismuth ( $n = 1.25$  to  $1.30$ ) [7], and for the organic conductor  $(\text{TMTSF})_2\text{PF}_6$  ( $n = 1.25$  to  $1.50$ ) [18].

To better characterize the transition from a metal to an insulating state induced by an applied magnetic field, the scaling theory proposed by Das and Doniach [5] was used. This theory implies the existence of Cooper pairs tightly bound and without long-range order, thus featuring a metallic state at absolute zero (Bose metal). Within this context, these authors proposed a scaling law used to characterize the transition from Bose metal to insulator based on two scaling parameters  $V_{ez}$ . In the inset the result of this scaling to a critical field  $H_C = 5.59$  T and critical exponent  $V = \frac{4}{3}ez = 1$  are shown.

The deviation of the Fermi liquid behaviour and a perfect correlation between the magnetoresistance data with the model proposed by Das and Doniach [5] suggests that, at low temperatures and zero applied magnetic field, the  $K_{0.05}\text{MoO}_{2.8}$  compound shows the expected for a Bose metal. In this context one may suggest a possible correlation between the unconventional metallic behaviour observed in the curves  $R(T)$  below  $\sim 70$  K with the existence of Cooper pairs tightly bounded without long-range order. This seems to corroborate with the observation of superconductivity in the  $K_x\text{MoO}_{2.8}$  system [10-11,15].

## 2. Conclusions

This chapter reported the influence of defects in the crystalline structure on the properties of K-doped  $\text{MoO}_2$ . Potassium doping leads to superconductivity with critical temperatures up to 10 K and electrical resistance measurements displayed an anomalous behaviour below the temperature showing the manifestation of magnetism ( $T < 70$  K). The study of the anomalies in the electrical behaviour provided important conclusions, such as the temperature dependence of the electrical resistance as described by a power law of the type  $R(T) = R_0 + CT^\alpha$ , where anomalous exponent ( $\alpha$ ) is found to be near 0.5. This value of  $\alpha$  described by the theory of Luttinger liquid (LL) suggests a mechanism for one-dimensional metallic conduction. Furthermore, the LL theory suggests that the interaction between the conduction electrons is attractive to positive  $\alpha$  and smaller than one. Under an applied magnetic field the electrical resistance changes to higher values indicating positive magnetoresistance. This behaviour is very similar to a Bose metal-insulator transition. This behaviour was interpreted according to the scaling theory proposed by Das and Doniach. This theory implies the existence of Cooper pairs tightly bound without long-range order, thus featuring a metallic state at absolute zero (Bose metal). This seems to agree with that observed by the LL theory and with the existence of superconductivity in the  $K_x\text{MoO}_{2.8}$  system.

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