Transport and magnetotransport properties of cold-pressed CrO$_2$ powder, prepared by hydrothermal synthesis

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Abstract

Submicron powder of CrO$_2$ was prepared by hydrothermal synthesis method from chromium trioxide with use of special modifiers, which govern the growth of particles. Particles obtained were of rounded form with mean diameter about 120 nm. The powder (stabilized with thin surface layer of $\beta$-CrOOH) has been characterized by structural, X-ray and magnetic measurements. The powder under investigation (with Curie temperature about 385 K) was cold-pressed and its transport and magnetotransport properties have been measured in the temperature range 4–450 K in magnetic field up to 1.6 T. The samples studied is characterized by non-metallic temperature behavior of resistance and large negative magnetoresistance (MR) in low temperature range. At $T = 5$ K the MR magnitude has been $-17\%$ at $H = 0.3$ T and $-20\%$ at $H = 1.4$ T. Its magnitude decreased fast with increase in temperature reducing to $0.3\%$ and less for $T > 200$ K. It is shown that this MR behavior is inherent for a system of magnetic grains with spin-dependent intergrain tunnelling. Some peculiarities of MR behavior in low-temperature range (below 40 K) can be associated with percolating character of tunnelling conductivity of this granular system under conditions of availability of only

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few conducting current paths through the sample.

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## 1. Introduction

Chromium dioxide ($\text{CrO}_2$) is ferromagnet ($T_C \approx 390$ K) which as a fine-particle medium has long been in use in the magnetic recording industry. This compound is predicted theoretically \[1\] to be a half-metal, that found experimental support \[2, 3, 4\]. In a half-metal, conductivity band at the Fermi level has carriers of only one spin type. Generally, spin polarization $P$ is determined as $P = (N_\uparrow - N_\downarrow)/(N_\uparrow + N_\downarrow)$ where $N_\uparrow$ and $N_\downarrow$ are densities of states for the up- and down-spin electrons, respectively. For half-metals, $P$ is expected to be equal 100% at low enough temperature what gives the opportunity for spintronics and nanotechnology devices.

Intrinsic magnetoresistance (MR) of crystal $\text{CrO}_2$ is known to be rather low (about 1%/T at room temperature) \[3, 4\]. At the same time, MR of the $\text{CrO}_2$ pressed powder with rather weak links between ferromagnetic (FM) grains can reach substantial magnitude (30% or more) at low temperature \[2, 5\]. This MR is extrinsic, that is determined by employed technology of preparation of granular material with insulating thin layers between FM grains. Insulating interlayers precludes direct FM exchange interaction between neighboring grains permitting, however, electron tunnelling. This tunnelling depends on the relative orientation of the magnetization in adjacent grains and is very sensitive to applied magnetic field. Tunnelling probability is maximal when moments in neighboring grains are aligned \[4, 6\]. MR of this type is called tunnelling MR (TMR) with MR mechanism being, so called, spin-dependent tunnelling \[4, 6\]. Significant spin polarization is the necessary prerequisite for high TMR.

Previous MR studies of the $\text{CrO}_2$ compacted powders were carried out mainly for commercial powders used for magnetic recording, which consists of acicular particles \[2, 3, 5, 7, 8\]. The powders were coated with thin surface layer of antiferromagnetic $\text{Cr}_2\text{O}_3$, which provides tunnelling contact between particles and, therefore, high enough TMR. It is of interest to study MR properties of $\text{CrO}_2$ powders prepared by alternative procedures and coated with insulating surface layers of different type. In this study, $\text{CrO}_2$ powder was...
prepared with hydrothermal synthesis, and CrO$_2$ particles were stabilized by converting surface layer of material into $\beta$-CrOOH. The cold-pressed samples studied consist of rounded particles of CrO$_2$. This ensures lesser shape anisotropy and porosity of samples. Results of measurement of transport and magnetotransport properties of the CrO$_2$ pressed powder are presented below.

2. Preparation and characterization of the chromium dioxide powder

In this work, the synthesis of CrO$_2$ has been carried out by hydrothermal synthesis method from mixture of 250 g of high-purity chromium trioxide (CrO$_3$), 50 g of distilled water and some special modifiers, which govern the growth of particles [9]. The synthesis was performed in an autoclave from stainless steel. Reagents have been put into test-tube of SIMAX glass. Maximum temperature of synthesis has been 320°C under pressure of 32 MPa. The resulting compound was dried in air at 150°C and milled in mill (MRP-2) with rotating knives. The powder obtained has been stabilized (by converting surface layer of material into rhombohedral $\beta$-CrOOH, chromium oxyhydroxide) in one liter of 0.3 M neutral solution of sodium sulfite under continuous stirring during 30 min. After that the magnetic powder has been extracted from stabilizing solution, washed with 2 liters of distilled water up to decolorization of the rinsing water. The paste obtained has been dried during 2 hours in desiccator at 150°C. The dried substance has been milled again.

The stabilized powder has specific surface equal to 10.5 m$^2$/g and consisted of closely sized rounded polycrystalline particles with mean diameter about 120 nm. The mean thickness of the stabilization surface layer of $\beta$-CrOOH is about 1 nm. This layer is insulating and non-ferromagnetic. Electron micrograph of the CrO$_2$ powder, obtained in transmission electron microscope JEM-100C, is shown in Fig. 1. In addition, the powder has been characterized with the X-ray and magnetization studies. Lattice parameters obtained are $a = 0.4419$ nm and $c = 0.2914$ nm in rutile-like lattice that agrees well with known data for pure CrO$_2$ [3]. The admixture components and $\beta$-CrOOH cause only slight shouldering of diffraction lines on the small-angle side, since admixtures have rutile-like structure and $\beta$-CrOOH is formed topotactically from CrO$_2$. Due to slight lattice mismatch $\beta$-CrOOH is
somewhat distorted. The stabilizing $\beta$-CrOOH layer in our synthesis method has orthorhombic lattice, same as was found in Ref. [10].

The powder obtained was pressed in a hydraulic press at 5 MPa to tablets with dimensions $3 \times 5 \times 10$ mm$^3$. Density of tablets consists 60% of the full X-ray density. Magnetic properties were measured in vibrating sample magnetometer (77 Hz). Examples of temperature dependences of the magnetization are shown in Fig. 2. The Curie temperature, $T_C$, was found to be $T_C \approx 112^\circ$C (385 K). At room temperature the compacted powder has coercive force about 0.0149 T, specific magnetization at $H = 1$ T about 62.5 Am$^2$/kg, remanent magnetization $M_r \approx 14.2$ Am$^2$/kg. At liquid nitrogen temperature (77 K), magnetization at 1 T increases up to 110.5 Am$^2$/kg, which can be taken as a low estimate for saturation magnetization $M_s$ (at liquid-helium temperatures range the specific magnetization should be evidently somewhat greater). It is known [3] that $M_s \approx 133$ Am$^2$/kg for pure CrO$_2$ that corresponds to magnetic moment per formula unit about 2 $\mu_B$. This value is larger then that measured in this work, which can be partly determined by the stabilization interlayers of non-magnetic $\beta$-CrOOH in the sample studied. Porosity determines internal demagnetization fields [11], that leads to rather low ratio $M_r/M_s$ in the sample studied, which is about 0.23 at room temperature taking the above-mentioned magnitude of magnetization at $H = 1$ T as $M_s$.

3. Transport and magnetotransport properties

3.1. General characterization

Transport and magnetotransport studies have been performed for two tablets obtained as indicated above. It is found that both tablets have essentially equal properties. Resistance as a function of temperature and magnetic field (up to 1.6 T) was measured using a standard four-point probe technique in a home-made cryostat. Current-voltage characteristics were linear for current below 0.2 mA. The measurements have been carried out in this low-current range where Ohm’s law is obeyed. Temperature curves for the resistivity are shown in Fig. 3. The curve 1 was recorded on heating of the as-prepared sample from 5 K to 430 K (after preliminary cooling down to 5 K in zero field). Resistance has strong non-metallic behavior ($dR/dT < 0$) and changes by nearly two order of magnitude in this temperature range. Nevertheless, $R(T)$ dependence is close to exponential one only at low tem-
perature \( T \leq 20 \text{ K} \). Narrowness of the temperature range does not permit us to determine exact form of this dependence.

Non-metallic behavior of \( \rho(T) \) and high value of \( \rho \) (as compared with that of pure CrO\(_2\) for which according to Ref. 5, residual resistivity is about \( 10^{-5} \text{ \( \Omega \) cm} \)) indicate that resistivity is determined by tunnelling of charge carriers between grains of CrO\(_2\) separated by \( \beta\)-CrOOH layers. In this case high TMR can be expected and is found. Magnetoresistance, \( [R(H) - R(0)]/R(0) \), measured in the field direction perpendicular to the current has appeared quite large (-20\% at \( T = 5 \text{ K} \) for \( H = 1.2 \text{ T} \), see Fig. 4) that agrees with that reported by other authors for CrO\(_2\) powder compact [2, 3, 5, 7, 8]. There are reasons to believe that such giant magnitudes of MR are determined by magnetic tunnelling between grains with high spin polarization [4, 6]. Extrinsic MR of this type is observed also in other compounds with high spin polarization of charge carriers, for example in mixed-valence manganites [2, 4, 12].

Specific feature of TMR is its rather rapid decline with temperature [2, 3, 8, 12], found in this study as well (Fig. 4). We have found that the temperature dependence of MR of the sample at \( H = 1.2 \text{ T} \) is described well by relation \(-[R(H) - R(0)]/R(0) \propto \exp(T/T_{mr})\) where \( T_{mr} \approx 45 \text{ K} \) (Fig. 4), in good agreement with results of other studies of CrO\(_2\) compacts [2, 3, 8]. This characteristic feature of the TMR is usually connected with strong decrease in polarization \( P \) with increasing temperature [6, 12, 13]. Other possible reasons of rapid decrease in MR of half-metallic granular systems with increasing temperature are considered in Refs. [1, 5, 6, 8].

During the measurements in the cryostat, the samples were kept rather long time in vacuum \( \approx 10^{-5} \text{ Torr} \). Apparently for this reason, the samples have appeared as unstable to heating above room temperature. Replicated measurements of \( \rho(T) \) after heating to 430 K show a considerable decrease in resistivity (Fig. 3) and MR (Fig. 4). More careful study has revealed that irreversible changes in the resistivity begin at temperature which is not so far above room temperature. It is evident that these changes in resistive and magnetoresistive properties are determined by eroding of intergrain stabilization \( \beta\)-CrOOH layers, and formation as well as strengthening of direct electrical interconnections between the CrO\(_2\) grains. The eroding is evidently accelerated when samples were placed in a rather high vacuum, since in air a significant eroding is not expected below 150\(^\circ\)C. A quite possible reason for this is that in vacuum the chromic acid residual liberates water in an accelerated way. This causes formation of CrO\(_2\) and, therefore, localized thinning of
stabilizing layer and occurrence of local short-circuiting bridges, which lead to decrease in the resistivity and MR. Stability of the $\beta$-CrOOH layer and mechanisms of its degradation need further research.

3.2. Field dependences of resistance

Magnetic-field dependences of resistance of the samples studied reflect their magnetic and structural properties in line with the suggested tunnelling character of conductivity and MR. Curves $R(H)$ have been recorded for different fixed temperatures according to the pattern similar to that used for routine measurements of hysteresis cycles of the magnetization. Initially, the field was increased up to $H_{\text{max}}$ (1.3–1.6 T). Thereupon $R(H)$ curves were taken in subsequent cycles of $H$ between $\pm H_{\text{max}}$, as shown in Figs. 5–8 for as-prepared state of the sample. The $R(H)$ curves were hysteretic with hysteresis enhancing dramatically with decreasing temperature (Figs. 5–8). For further discussion of the hysteresis phenomena we shall distinguish curves $\Delta R(H)/R(0)$ by direction of the field sweeping. The first type MR curves are taken with changing field from $+H_{\text{max}}$ to $-H_{\text{max}}$; whereas, the second type curves are recorded with field sweeping in the opposite direction, from $-H_{\text{max}}$ to $+H_{\text{max}}$. We denoted these types of MR curves, respectively, by $\text{MR}(\leftarrow)$ and $\text{MR}(\rightarrow)$ as shown in Figs. 5 and 7.

In low-field range, all MR curves show two not very high peaks of positive MR, which are symmetrically positioned about $H = 0$. These peaks of approximately the same height are positioned at fields $H = H_p$ and $H = -H_p$ (position of $H_p$ is shown in Figs. 5 and 6). With increasing temperature the peaks become rather flat (see Fig. 8 for $T = 50$ K). Such type of hysteresis is generally expected for TMR of half metallic granular samples. It is believed [4, 6, 13] that MR curves in this case reflect magnetization hysteresis cycles so that resistance is expected to be maximum at demagnetized state of the sample at zero magnetization, which takes place at $H = H_c$ or $-H_c$, where $H_c$ is coercive force. So that the relation $H_p \approx H_c$ is expected, which was really found for some half-metallic systems including CrO$_2$ powder compacts [4, 5]. As magnetization increases with increasing field, mutual alignment of magnetic moments in adjacent grains becomes stronger resulting in an increase in MR. It can be expected, therefore, that, when $M$ goes close to the saturation value $M_s$, MR will go to some saturation limit as well. This behavior seems to be true when sample was studied at low enough temperature (Fig. 5).
For the samples studied, the simple correlation \([4, 6, 13]\) between hysteretic behaviors of \(R(H)\) and \(M(H)\) appears to be valid only for high enough temperature (Figs. 7 and 8). Here MR curves taken for different sweep directions, \([\text{MR}(\leftarrow) \text{ and MR}(\rightarrow)]\), merge together at a high enough field like corresponding hysteretic \(M(H)\) curves. At low temperature (below 15 K), however, an additional hysteresis effect (crossing of the MR(\(\leftarrow\)) and MR(\(\rightarrow\)) curves) is clearly seen for fields not far above \(|H_p|\) (Figs. 5 and 6) which is inconsistent with the simple model picture for a single magnetic tunnel junction. It is no surprise that behavior of the sample studied, which presents an array of tunnel junctions, differs from that expected for single junction.

It is known that quantum tunnelling of the charge carriers occurs between states of equal energy. Actually, however, there is always some energy level mismatch between electron states in neighboring grains for different reasons. In this case, a charge carrier should gain some energy (for example, from phonons) to accomplish the tunnelling. The intergrain conductivity is conditioned, therefore, by the two processes: the tunnelling and thermal activation.

In real granular metals, thickness of insulating interlayers between grains is not the same throughout the system, thus, the conductivity is percolating \([14]\). It is determined by the presence of “optimal” chains of grains with maximum probability of tunnelling for adjacent pairs of grains forming the chain. In conditions of activated conductivity, number of conducting chains decreases with decreasing temperature, so that at low enough temperature a percolation network can even come to a single conducting path \([14]\). These “optimal” chains have some weak links (high-resistance tunnel junctions) with increased activation energy, that cannot be avoided. These high-resistance junctions, in fact, determine the activated character of total measured conductivity.

Picture of tunnelling conductivity in granular half-metallic systems is more intricate than that in non-magnetic systems. Here the tunnelling probability depends not only on properties of insulating intergrain barriers but also on the mutual alignment of magnetic moments in neighboring grains. It is clear, therefore, that at a fixed temperature the spacial positions of the “optimal” chains of grains with maximum conductivity (and the positions of high-resistance junctions within chains, which determined the whole system resistance) are continuously changing with variation of external magnetic field. Moreover, application of an external magnetic field induces opening of additional transport channels \([15]\) that causes giant MR in such percolating
systems. The additional MR hysteresis (Figs. 5 and 6) in the field range above $H_p$ is evidently takes place only in conditions of small number of percolating current channels at low enough temperature. This can indicate that varying percolating sets of current channels are different for increasing and decreasing magnetic field in this field range.

It is generally believed [14] that activation energy is mainly determined by Coulomb charging energy $E_C = e^2/\kappa d$, where $\kappa$ is effective dielectric constant and $d$ is average grain diameter. Taking $\kappa = 5$ for CrO$_2$ compact powder (as that in Ref. [5]) and $d \approx 120$ nm, a charging energy for the sample studied is about 28 K. It is not surprising then that, as indicated above, $R(T)$ dependence is close to exponential one only for $T < 20$ K. In any case, rising temperature leads to increase in effective number of current paths through the sample and, therefore, to more homogeneous current distribution within the sample [14, 15]. The conductivity in magnetic field is determined in this case by magnetic state of far more number of grains, than in the case of few current paths at low temperature, so that hysteretic behaviors of $R(H)$ and $M(H)$ are mutually correlated at fairly high temperature in the expected way.

Occurrence of the additional MR hysteresis at low temperature range (Figs. 5 and 6) is accompanied by peculiar temperature behavior of characteristic field $H_p$ (which is point of maximum resistance in the MR hysteresis cycle). With increasing temperature $H_p(T)$ first rises and then (above 30 K) goes down (Fig. 9). Decrease in $H_p$ with decreasing temperature below 30 K is unexpected under common suggestion $H_p \approx H_c$.

At first glance the sample studied can be considered as a system of single-domain grains with weak exchange coupling between the grains. Really, the critical single-domain diameter of spherical particles for CrO$_2$ is estimated to be about 200 nm [16] which is larger than average grain size (120 nm) in the sample studied. However, due to rather wide grain-size distribution and other reasons it cannot be excluded that some grains are multidomain. It can be seen in Fig. 1 that certain of the particles are polycrystalline. It follows from Fig. 1 as well that some of the small particles can stick together rather strongly and, after stabilization by $\beta$-CrOOH, they can make multidomain grains. Consequently, the sample studied can be a mixture of single-domain and multidomain grains.

In an isolated single-domain grain, increase in applied field above some nucleation field $H_N$ leads to rotation of the magnetization out from the easy axis into the direction of the applied field (this mechanism of magnetizing is
called homogeneous rotation) \[16\]. For spherical particle \( H_N = -2K_1/M_s \), where \( K_1 \) is the anisotropy constant. Field \( H_N \) determines upper limit for coercive force \( H_c \). \( K_1 \) is strongly temperature dependent (reduces with increasing temperature more rapidly than \( M_s \) \[17, 18\]), and goes to zero at the Curie temperature. Typical behavior of \( H_c(T) \) is suggested to be the same.

The coercive force \( H_c \) in real granular systems is usually much smaller than \( H_N \) for a single particle due to different sources of inhomogeneities and dependence of \( H_c \) on the strength of intergrain exchange coupling. To include these effects, a general relation for the coercive fields of real granular ferromagnet is used \[16\]:

\[
H_c = \alpha(2K_1/M_s) - N_{eff}M_s,
\]

where numerical parameter \( \alpha (< 1) \) takes into account the microstructure disordering and \( N_{eff} \) depends on local demagnetization fields of grain edges.

The coercive force \( H_c \) is a field at which a reversal of the magnetization takes place when, after magnetizing of a sample for one field direction, an increasing magnetic field is applied in an opposite direction. Magnetic particles of sufficiently large size will generally not be uniformly magnetized but rather be composed of magnetic domains. Consequently, in multidomain particles, the magnetization reverse can be reached not only with uniform rotation of magnetization but more easily with nucleation and growth of domain with opposite direction of magnetic moments \[11, 17, 18\]. For this reason, value of \( H_c \) in multidomain particles is far less than that in single-domain ones.

For homogeneous (single-domain or multidomain) powder systems, \( H_c \) is expected to be maximal at low temperatures but should decrease with increasing temperature going to zero at \( T \simeq T_C \). The field \( H_p(\approx H_c) \) follows this expected behavior, but only above 30 K (Fig. 9). Position of a point of \( H_c \), obtained from magnetization measurement at room temperature, fits in this behavior (Fig. 9). However, decrease in \( H_p \) with decreasing temperature below 30 K (Fig. 9) is unexpected in a fairly homogeneous powder systems and is evidently closely related with peculiar inhomogeneous structure of the system studied and percolating character of its conductivity. In granular systems at low enough temperature there are only a few conductive current paths. These are “optimal” chains of grains with maximum conductivity, and it is very likely that they consist preferably of grains of larger size. Such grains are multidomain with smaller coercive force \( H_c \) that can explain the surprising behavior of \( H_p(T) \) in low temperature range (Fig. 9).

In conclusion, we have studied cold-pressed powder of \( \text{CrO}_2 \), prepared by hydrothermal synthesis, with mean diameter of particles about 120 nm. The powder was stabilized with thin surface layer of \( \beta\text{-CrOOH} \). Transport and
magnetotransport properties of the sample correspond to behavior of magnetic granular system with weak exchange interaction between the grains. Large MR in low temperature range can be well ascribed to spin-dependent intergrain tunnelling. Some peculiar properties of MR in low-temperature range \( (T < 40 \text{ K}) \) can be sure attributed to percolating character of tunnelling conductivity of this granular systems in conditions of few current paths through the sample.

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Figure captions

Figure 1. Transmission electron micrograph of the CrO$_2$ powder.

Figure 2. Temperature dependences of magnetization for different external fields. The dependences were recorded on heating, after the sample had been cooled down from the room temperature to $T = 77$ K in zero field.

Figure 3. Temperature dependences of the resistivity for different states of the sample (see main text).

Figure 4. Temperature dependences of the magnetoresistance $\Delta R(H)/R(0) = [R(H) − R(0)]/R(0)$ at $H = 1.2$ T for as-prepared state of the sample (○) and after heating the sample up to 430 K (●). The inset shows the dependence for the as-prepared state in semilogarithmic plot, demonstrating exponential temperature dependence of MR below 200 K.

Figure 5. (Color online) (a) MR curves at $T = 5.1$ K recorded with magnetic field variation $H_{\text{max}} \rightarrow -H_{\text{max}}$ [MR(←)] and $-H_{\text{max}} \rightarrow H_{\text{max}}$ [MR(→)], where $H_{\text{max}} \approx 1.5$ T. Inset shows blowup of the MR behavior in low-field range. Peak of positive MR at field $H_p$ is indicated by arrow. (b) Magnetic-field dependence of difference between curves MR(→) and MR(←) taken for opposite directions of the magnetic-field sweeping.
Figure 6. (Color online) MR curves recorded at $T = 7.04$ K. Measurement protocol is the same as that described in capture to Fig. 5. Inset shows blowup of the MR behavior in low-field range.

Figure 7. (Color online) (a) MR curves at $T = 15.04$ K recorded with magnetic field variation $H_{\text{max}} \rightarrow -H_{\text{max}}$ [MR(←)] and $-H_{\text{max}} \rightarrow H_{\text{max}}$ [MR(→)]. Inset shows blowup of the MR behavior in low-field range. (b) Magnetic-field dependence of difference between the MR(→) and MR(←) curves taken for opposite directions of magnetic-field sweeping.

Figure 8. (Color online) MR curves recorded at $T = 50$ K. Inset shows blowup of the MR behavior in low-field range.

Figure 9. Temperature dependence of magnetic field $H_p$ at which resistance is maximum in MR curves. Symbol ▲ indicates the position of coercive force $H_c$ at room temperature, obtained in magnetization measurements. The data are related to as-prepared state of the sample studied.
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