Large magnetoresistance effects in Fe₃O₄

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Received 22 November 2018, revised 18 February 2019
Accepted for publication 5 March 2019
Published 26 March 2019

Abstract
We investigated the magnetoresistance (MR) of a single crystal of magnetite, Fe₃O₄. In an effort to distinguish between different contributions to the MR the samples were prepared in two different initial magnetic states, i.e. by either zero-field or by field cooling from room temperature. The different magnetic structures in this sample have a dramatic effect on the magnetoresistance: for initially zero-field-cooled conditions a negative MR of about −20% is observed just below the Verwey transition at \( T_V \approx 124 \) K. For decreasing temperature the MR increases, changes sign at \( \sim 78 \) K and reaches a record positive value of \( \sim 45\% \) at around 50 K. This behavior is completely absent in the field-cooled sample. Magnetization measurements corroborate an alignment of the easy magnetization direction in applied magnetic fields below \( T_V \) as a cause of the strong effects observed in both, magnetization and MR. Our results point to a complex interplay of structural and magnetocrystalline effects taking place upon cooling Fe₃O₄ through \( T_V \).

Keywords: magnetite, magnetoresistance, magnetization, magnetocrystalline effects

(Some figures may appear in colour only in the online journal)

1. Introduction
Magnetite (Fe₃O₄) as the first known magnetic material has traversed history as a room temperature ferrimagnet of interest in applied magnetism, mineralogy, biology, and geophysics. Even today, it attracts considerable scientific and technological interest for its applications in palaeomagnetism [1], magnetic sensors [2], catalysis [3] and nanomedical carriers [4]. Especially the high Curie temperature [5] of \( T_C \approx 860 \) K and the theoretically predicted half-metallic character [6] make Fe₃O₄ very attractive for room temperature spintronic applications [7, 8]. In addition, magnetite is a parent compound for magnetic materials such as maghemite and spinel ferrites. Besides its unique magnetic properties, the main interest was focused on the mechanism and physical consequences of the first-order metal-insulator phase transition, known as the Verwey transition [9]. This discontinuous jump in transport properties is accompanied by a transition from a cubic \( Fd\bar{3}m \) to a monoclinic \( Cc \) structure type below \( T_V = 124 \) K [5, 9–18].

Although Fe₃O₄ has been widely studied, there is only little known about the magnetoresistance (MR) of the bulk material. Bulk Fe₃O₄ has been reported to exhibit very small positive or negative MR, depending on the magnitude of the applied magnetic field, the crystallographic orientation, and the magnetic history of the sample at temperatures below \( T_V \) [19–27]. Moreover, a minimum MR at \( T_V \) for bulk magnetite is also observed, which is proposed to be caused by either the field-dependent discontinuous entropy change [25] or by the extreme sensitivity of the mixed state in the transition region (order-disorder of Fe²⁺ and Fe³⁺ ions) to an applied magnetic field resulting from coupled phonon–magnon states [28]. However, the MR results of previous work were often obtained on natural magnetite with slightly varying composition [29] and the influence of the magnetic sample history on the MR below \( T_V \) has not been studied, yet. These conditions are expected to significantly influence the different electronic orderings. There is an argument of orbital ordering through a continuous phase transition along with the spin reorientation,
i.e. an interplay between the electronic degrees of freedom, which appears to be unaffected by the Verwey transition [30, 31]. These electronic degrees of freedom may contribute to the MR in a very diverse fashion. Moreover, there is a change of the easy magnetization direction (EMD) at a temperature \( T_K \approx 130 \text{ K} \) distinct from \( T_V \) [13].

Here, we systematically investigated the MR effect in a high-quality single crystal of Fe₃O₄ at low temperatures. Below the Verwey transition we observed a substantial effect of the magnetic history of the sample on the measured MR as well as a very large MR of up to 45\%, which is about an order of magnitude larger than the generally reported values for bulk Fe₃O₄ [23, 25, 27]. Moreover, a sign reversal of the MR at around 78 K is observed. By controlling the magnetic history of the sample the interplay of crystallographic and magnetic structure as well as the influence of magnetocrystalline anisotropy on both, the MR and the magnetization, is qualitatively revealed.

2. Experimental

The Fe₃O₄ single crystal was grown by the floating zone technique [32]. The quality of our sample was examined by powder and single crystal x-ray diffraction and Laue diffraction at room temperature, as well as \( T \)-dependent transport and magnetic properties measurements. A high value of \( T_V \approx 124 \text{ K} \) indicates the high sample quality [29, 33–39]. The single crystal was cut into a bar-shaped cuboid with dimensions of 5.7 mm [100] \( \times 2.0 \text{ mm } [010] \times 0.9 \text{ mm } [001] \). Throughout this article, crystal axes of [100], [010], [001], and [111] refer to the high temperature cubic structure. The magnetic properties were measured by using a magnetic property measurement system (MPMS, Quantum Design Inc.). The transport measurements, which were performed by standard four probe technique, and the heat capacity measurements were conducted by using a physical property measurement system (PPMS, Quantum Design Inc.). The temperature dependent resistivity \( \rho(T) \) was measured in zero-field cooling (ZFC) or field cooling (FC) conditions by applying an electric current of \( I = 1 \mu \text{A} \) and ramping the temperature from 300 K to 44 K (and back to 300 K). The magnetoresistance and magnetization measurements were conducted at different temperatures below \( T_V \) in two different ways: (A) for each target temperature the sample is zero-field cooled from 300 K and subsequently \( \rho(H) \) or \( M(H) \) is measured as a function of magnetic field up to 50 kOe, and (B) the sample is initially field cooled in 50 kOe from 300 K to 124 K, i.e. cooled in applied field through \( T_K \) to the Verwey transition, and then \( \rho(H) \)- or \( M(H) \)-curves are measured at constant \( T \). Subsequently, the temperature is reduced for the next \( \rho(H) \)- or \( M(H) \)-measurement without prior warming-up.

3. Results and discussion

Figure 1(a) presents the ZFC magnetoresistance at different temperatures and for measurement condition (A). Here, the magnetic field \( H \) as well as the electric current \( I \) were applied parallel to the crystallographic [100] sample direction, see the inset of figure 1(d). At temperatures just below \( T_V \) a relatively large negative magnetoresistance MR = \( \rho(H) - \rho(0) / \rho(0) \) of up to \(-20\% \) is observed. This value of MR is seen already at small fields, but decreases only very little as the field is ramped up to 50 kOe. Upon decreasing temperature, the MR value slightly varies to about \(-15\% \) at 90 K and then rapidly increases, thereby changing sign from negative values for \( T > 80 \text{ K} \) to positive values for \( T < 75 \text{ K} \). At 47 K, it reaches a substantially enhanced value of \(+45\% \). We note that these effects have not been reported before [19–27]. In contrast, the MR values obtained for measurement condition (B) and shown in figure 1(b) are negative for all \( T < 124 \text{ K} \) and comparably small (MR < 10\%), similar to the values reported in literature [23, 27]. Clearly, applying ZFC condition (A) is essential for the observation of enhanced MR values.

Although bulk Fe₃O₄ is ferrimagnetic below \( T_C \sim 860 \text{ K} \), the EMD (hard magnetization direction) changes from [1 1 1] ([100]) above \( T_K \) to [0 0 1] ([111]) for temperatures below \( T_K \) [40–42]. Under FC condition (B) the EMDs can be aligned with the field direction upon cooling through \( T_V \). In contrast, under ZFC condition (A) small magnetic domains of different orientations of the EMDs in the monoclinic phase are formed. In this case the initial magnetization state at each measurement temperature below \( T_V \) can be considered as almost identical because the sample is nearly completely demagnetized by ZFC from well above \( T_K \) [43]. The initial magnetization processes upon application of the field required for the MR measurement then aligns these magnetic domains for fields higher than a critical one, \( H \sim H_{cr} \). The MR is greatly affected by the orientation of the moments in magnetic materials [44]. In addition, the domain walls contribute to the MR [45]; upon application of a magnetic field the number of domain walls is usually reduced (in the absence of strong pinning) through a growth of the magnetic domains. Such a behavior is in good agreement with the observed rapid decrease of \( \rho(H) \) at small fields \( H_{cr} \), the very small change of \( \rho(H) \) for larger fields \( H > H_{cr} \), and the constancy of the resistance after removal of the applied field in a remanent magnetization state for \( T > 80 \text{ K} \), see figure 1(c). It also explains the highly reduced MR for measurement condition (B) because here, the magnetic domains are already aligned during the field cooling, and the domain structure is not significantly altered between different measurement temperatures, figure 1(b). On the other hand, it does not provide a straightforward mechanism that accounts for the positive MR at lower \( T \).

When addressing the latter issue we need to keep in mind that the slight crystallographic distortion for \( T < T_V \) towards a monoclinic structure with a larger unit cell typically results in a multi-crystalline structure at low temperature. A single-crystalline structure can only be retained by simultaneously applying a magnetic field of 10 kOe upon cooling through \( T_V \) (which aligns the EMD, i.e. the crystallographic c' -axes that are perpendicular to the monoclinic a-b planes) as well as mechanically straining the sample in order to de-twinn the single crystal [46]. Such an elaborate procedure was not applied to our sample and hence, the sample should be considered twinned (FC, condition B) or even polycrystalline.
(ZFC, condition A) below \( T_V \). We also note that the magneto-crystalline anisotropy changes significantly at \( T_V \) [13, 46].

In general, the constants \( K_i(T) \) of magnetocrystalline anisotropy (where \( i \) indicates the crystallographic directions) are temperature dependent. An early study of \( K_i(T) \) revealed that the leading constant \( K_m^a \) in the monoclinic phase exhibits only a small temperature dependence and outweighs \( K_c^a \) of the cubic phase by about an order of magnitude [13, 47]. In consequence, the impact of the magnetocrystalline anisotropy on the resulting domain structure in the monoclinic phase is much larger [13, 47]. This jump in magnetocrystalline anisotropy can explain the comparatively small MR values obtained under condition (B), figure 1(d): in this case, the monoclinic phase is retained and the magnetocrystalline anisotropy energy stays high (compared to the cubic phase). Hence, the sample is not demagnetized, in contrast to the discussion above for condition (A). This prevents a major disarrangement of the magnetization alignment and/or redistribution of the EMDs as discussed below. In result, the MR exhibits very little temperature dependence once the magnetization has become aligned at the first few measurements (\( T > 115 \) K) at temperatures close to \( T_V \) [25, 27].

The impact of temperature on the crystalline and magnetic microstructure as well as the magnetization alignment is corroborated by magnetization measurements. Figure 2(a) exhibits the initial \( M(H) \)-curves at different temperatures after ZFC from 300 K, condition (A), in applied fields \( H \parallel [100] \) up to 50 kOe. In order to clearly visualize the variation of \( H_{cr} \) with temperature, the \( M(H) \)-curves are only presented in the field range 0–10 kOe. A jump of the magnetization at \( H_{cr} \) and an increase of \( H_{cr} \) with decreasing temperature is observed. For \( H < H_{cr} \), we interpret these curves by a progressive alignment of the domain magnetization against the magneto crystalline anisotropy. Such an assignment is heavily supported by the overlap of the \( M(H) \)-curves for \( H < H_{cr} \) at different temperatures given the above-mentioned small \( T \)-dependence of the magnetocrystalline anisotropy. However, this small temperature dependence of magnetocrystalline anisotropy cannot explain the strong increase of \( H_{cr} \) with decreasing temperature, particularly below about 80 K, see figure 2(b). Also, it seems unlikely that the substantial values of \( H_{cr} \) at these low temperatures can be merely caused by magnetocrystalline anisotropy [13] in Fe3O4, e.g. by irreversible magnetization reversal, given the small coercivity [43] of only a few ten Oe even at temperatures below \( T_V \). Rather, the polycrystalline nature of our sample in condition (A) should be taken into consideration: an applied field can align (or switch) the EMDs, i.e. the \( c^* \)-axes (which are perpendicular to the monoclinic \( a-b \) planes) of the individual crystallites [13, 40, 42, 47–49]. This alignment, which involves a field-induced electronic transfer,
appears to be thermally assisted, see inset to figure 2(b). By fitting ln($H_{cr}/T$) versus 1/$T$,[40, 42], an activation energy of about 25 meV is estimated, which compares favorably to the value reported by Kakol et al [42]. Note, however, that our measurement protocol is slightly different from this previous work in that the magnetic moments of the domains are not fixed to any directions when the sample is zero-field cooled from 300 K.

Obviously, under conditions (B) the sample remains in the monoclinic phase, i.e. the crystallographic and magnetic structure is largely maintained as we go lower in temperature, in a similar fashion as discussed for the MR. Particularly, the $c^*$-axes, i.e. the EMDs, are aligned due to the FC protocol. Hence, the magnetization can be very easily rotated within an applied magnetic field even at low $T$, inset to figure 2(a). Importantly, the $H_{cr}$-values obtained from magnetization and MR measurements agree very well if the field is applied in an identical direction ($H[100]$) which testifies that both quantities are governed by the same underlying mechanisms of magnetic and structural (i.e. EMD) alignment. Figure 2(a) then also provides insight into the decreased MR-value for $T < 47$ K: at these low temperatures our maximum applied field does not suffice to reorient the EMDs (i.e. the $c^*$-axes) of the monoclinic phase, in line with the findings of [42].

The question remains why we observe a reversal of the sign of the MR for measurement condition (A) upon lowering the temperatures. The negative MR just below $T_V$ (figure 1(c), curve at 100 K) can be understood by an alignment of the domains and a decimation of domain walls changing...
the system towards a more ordered state [44] as the EMDs align. It was pointed out by Kakol et al. that the reorientation of the EMD involves a re-arrangement of the ionic order, i.e. a structural change [42]. However, the positive MR for \( T \lesssim 78 \) K requires an additional mechanism. Application of a magnetic field does not allow to distinguish between a mere alignment of the magnetic moments against (small) magnetocrystalline anisotropy or a simultaneous alignment/switching of the underlying crystallographic axes [42, 50]. Also, field-cooling the sample and subsequent measurement of the MR with magnetic field applied in different directions with respect to the field used for FC did not show any of the large MR effects reported here nor indications of a sign change [42, 50]. Hence, ZFC condition (A) appears to be a prerequisite for our observation of significant MR as well as its sign change.

The magnetocrystalline anisotropy energy \( E_m^k \) in the monoclinic phase was introduced [13, 47] to contain—in addition to the contributions along the crystallographic \( a \) and \( b \) axes—a diagonal term (with a magnetocrystalline anisotropy constant \( K_m^u \) defined along the \([111]\) direction) due to the small rhombohedral distortion. This additional term retains the \( c' \)-axis as the EMD as long as \( K_m^u \leq 0 \) (note the negative sign in the definition of \( K_m^u \) [13, 47]). However, this may change for \( K_m^u > 0 \), and indeed \( K_m^u \) is reported to change sign from negative to positive upon cooling below about 80 K [13, 47]. For \( K_m^u > 0 \) the EMD may somewhat deviate from the \( c' \)-axis, particularly in the \( c'-b \) plane since \( E_m^k \) increases much less rapidly towards the \( b \)-axis as compared towards the magnetically hard \( a \)-axis. Such a deviation will cause twinning which in turn may explain the increasing MR. One may even speculate that \( K_m^u \) needs to overcome a minimum positive value for this effect to take place and hence, a somewhat lower temperature for the sign reversal of the MR (compared to the sign change of \( K_m^u \)) may be plausible. For \( T \) decreasing further, \( K_m^u \) increases while the anisotropy constant \( K_m^b \) decreases, i.e. the twinning becomes more pronounced, which may explain the increasing positive MR at lower \( T \), figure 1(d).

Before providing further support for this explanation of the sign reversal of the MR by changing the field orientation, we analyze the resistivity and magnetization for \( H \parallel I \) in more detail. Figure 3(a) shows the resistivity \( \rho(T) \) as a function of temperature for ZFC with \( H \parallel I \) [1100]. The high value of \( T_V \approx 124 \) K together with the steep jump in \( \rho(T) \) by more than two orders of magnitude and a narrow hysteresis of 0.3 K demonstrate the exceptionally high quality of our Fe3O4 single crystal [29, 37, 51]. We also measured the \( \rho(T) \)-curve...
for FC in 50 kOe. The right inset of figure 3(a) displays \( \Delta \log(\rho(T))/\Delta T \) in dependence on \( T \) for the ZFC and FC conditions. Interestingly, the difference between the derivatives at these two fields, \( \Delta \log(\rho(T))/\Delta T \), is largest at around 74 K, see left inset of figure 3(a).

In addition, the zero-field and field cooling \( M(T) \)-curves of FeO are compared in figure 3(b). A sharp jump in magnetization at \( T_V \approx 124 \) K and a small peak at \( T_K = 130 \) K, corresponding to the spin reorientation can be observed, in agreement with [13, 30]. Below \( T_V \), the magnetization in FC conditions is larger as in ZFC, as expected. The ZFC magnetization data are shown in more detail in the right inset of figure 3(b): a small anomaly is observed at \( T \approx 30 \) K which was reported before [52]. Surprisingly, another small hump at 78 K can be seen as well, a temperature that coincides with the one at which the MR changes sign, figure 1(d). Such a very small decrease of magnetization is consistent with a deviation of the EMD from the \( c' \)-axes below about 80 K. In contrast, these feature of the magnetic properties cannot be seen in the FC \( M(T) \)-curve (left inset of figure 3(b)). It should be noted that the \( T \)-dependence of the specific heat did not exhibit any anomaly at around 30, 78 or 130 K, see figure 3(c) indicating that there are no phase transitions involved at these particular temperatures. In case of the above-discussed small deviation of the EMD from the \( c' \)-axis below about 80 K no anomaly of the specific heat is expected around this temperature.

To further scrutinize our above interpretation of the magnetization behavior in FeO, we also measured the transport properties for field orientation \( H \perp I \). As shown in figure 4(a) there is again a large change in the resistivity with \( T \), and a jump at \( T_V \). Importantly, we also observe large values of MR, figure 4(c). In contrast to the field orientation \( H \parallel I \), however, the MR is now positive at \( T > 78 \) K and changes sign at lower \( T \). Usually, such a dependence of the MR on the orientation between \( H \) (and consequently \( M \) and \( I \)) is discussed within the framework of anisotropic MR (AMR). However, AMR effects in FeO bulk and thin films are reported [27, 53] to be much smaller and, importantly, are not expected to change sign well below \( T_V \), in contrast to our observation, figure 4(d). This clearly indicates the involvement of the crystallographic orientation: the orientation of the field determines the crystallographic orientation which, with respect to the current, is decisive for the sign of the MR. It confirms the alignment of the \( c'-\)axises, and hence of the magnetic moments, of the crystallites, which is in one case parallel to \( I \), and perpendicular to \( I \) in the other.

Furthermore, the derivative \( d \log(\rho)/dT \) in dependence on \( T \) between 50–120 K for FC condition in \( H = 50 \) kOe reaches a maximum at 74 K, as shown in figure 4(b) along with the derivative of \( \log(\rho) \) for ZFC condition. The difference between the two curves, \( \Delta(d \log(\rho)/dT) \), now displays a minimum at 74 K (see inset of figure 4(b)). Note that the same temperature was inferred from \( \rho(T) \) for \( H \parallel I \), figure 3(a), yet with opposite sign of the difference \( \Delta(d \log(\rho)/dT) \) (see left inset to figure 4(a)).

Albeit our combined investigation of magnetotransport and magnetization for different magnetic field orientations and histories provides clear signatures for an influence of the switching of the EMD and the magnetocrystalline anisotropy on the afore-mentioned properties, we discuss possible alternative interpretations in the following. Lee et al reported a positive MR of a weakly disordered electron gas arising from spin splitting of the conduction-electron energies [54]. In result, the MR scales as \( H^2 \) for small magnetic fields, and \( \sqrt{H} \) for large fields, a behavior not observed in our FeO. Also, spin–orbit scattering from paramagnetic impurities in nonmagnetic metals [55] (the so-called weak antilocalization effect), as well as the quantum electron-electron interference effect, or a variation of the Fermi surface in applied magnetic field [56] may give rise to a positive MR. These effects, however, appear unlikely to explain our results.

The conductivity in FeO for \( T < T_V \) is usually discussed in terms of variable range hoping of small polarons [57]. Yet, even below \( T_V \) alternative mechanisms have been discussed for different temperature ranges, see e.g. [58, 59]. In principle, a change in the transport mechanism may also cause changes in the MR. The large effects and orientation dependencies observed here, figures 1(d) and 4(d), however appear to render such an explanation unlikely.

On more general grounds, a reversal of the MR near a phase transition has been experimentally reported in many materials, see e.g. [60–63]. However, in these studies the MR is reported to exhibit a quadratic or/and linear magnetic field dependence [61–63], which is obviously different from our results, see figure 1(a).

4. Conclusion

In summary, we have systematically studied the MR and the magnetization in high quality single crystals of FeO below the Verwey transition. Under ZFC conditions we observe an extraordinarily large MR of up to +45% and a sign reversal of the MR at ~78 K. Magnetization measurements reveal the alignment of the easy magnetization direction in applied magnetic field, i.e. an interplay of structural and magnetic properties. The temperature dependence of the critical magnetic field for this alignment of the EMD is almost identical to the one obtained from MR implying an identical underlying mechanism. The MR strongly depends on the field orientation, and hence the magnetization orientation, with respect to the current applied for MR measurements which may be crucial in case of future applications. The large MR is only observed if the sample is zero-field cooled through the structural transition at \( T_V \), i.e. it is absent if the sample remains in the monoclinic phase. Consequently, both MR and magnetization are governed by changes of the crystallographic and magnetic microstructure. Our observations therefore highlight the intricate interplay of structural and magnetocrystalline anisotropy changes near \( T_V \).

Acknowledgments

We thank W Liu and Z D Zhang from the IMR CAS for helpful discussions. This work has been supported by the Max Planck-POSTECH Center for Complex Phase Materials.
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