Possible evidence for a spin-state crossover in the Verwey state in Fe_3O_4 thin films

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In epitaxial thin films of magnetite a large change in magnetization across the Verwey transition has been observed. In the Verwey state, the sample magnetization appeared to be strongly reduced, in some samples even close to zero. Using superconducting quantum interference magnetometry, x-ray absorption near edge spectroscopy, and polarized neutron reflectometry, a simple rotation of the magnetization vector due to a change in magnetocrystalline anisotropy was excluded. The experimental data rather suggest an intrinsic loss of magnetic moment due to a possible transition into a low or intermediate spin state of Fe^{2+}. This observation discloses a different aspect of the Verwey transition.

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The first magnetic material known to mankind, magnetite (Fe_3O_4), has a rich history, spanning from being used in a compass to being considered as a potential material for the emerging field of spintronics. In solid state physics, however, it has been the Verwey transition that has puzzled and intrigued scientists all over the world since its discovery [1]. From the very beginning of the investigation of the Verwey transition it has been considered as one of the most prominent examples of charge ordering [2,3]. In the recent literature, numerous authors suggest a complex charge and orbitally ordered state associated with a phase transition into a monoclinic structure [4]. The details of this charge and orbitally ordered state as well as the driving force behind it remain a matter of ongoing debate [5–14]. In most of the experimental data, the magnetization evolution through the Verwey transition shows a feature of varying characteristics. Mainly, a relatively sharp drop of magnetization of varying size has been reported for almost all magnetite samples [15,16]. Due to its dependence on the orientation of the applied field with respect to the magnetite crystal axes this phenomenon sometimes has been attributed to the change of magnetic easy and hard axes when the room-temperature $Fd\bar{3}m$ (space group No. 227) cubic symmetry turns into a lower crystal symmetry [17,18]. Using nuclear magnetic resonance (NMR) a magnetic rotation temperature of $T_{mr} = 126$ K was determined which is slightly above the Verwey transition [18]. A study combining superconducting quantum interference magnetometry (SQUID) and magnetic force microscopy (MFM) has shown that the domain pattern remains unchanged across the Verwey transition. The authors suggested that the magnetic moment has a tendency to rotate out-of-plane in the Verwey state, but only by a relatively small rotation angle of less than 10° [19]. Note that the magnetic moment decreases for all directions of the magnetic field applied parallel to the three principal axes of the MgO substrate [19]. The models used to describe the Verwey transition mostly do not consider a possible *intrinsic* change in magnetization, e.g., a transition into a low-spin state of the Fe^{2+} $3d^6$ state. Such a crossover might also occur at the Fe^{3+} $3d^5$ site. Due to the antiferromagnetic coupling of the Fe^{3+} moments, this crossover is not expected to be associated with a change in magnetization. It is important to note that the experimentally observed change in magnetic anisotropy is not sufficient to explain the almost vanishing magnetic moment in the Verwey state reported here. In particular, the use of thin films with a very strong shape anisotropy should further reduce the influence of the intrinsic magnetocrystalline anisotropy. Using SQUID, x-ray circular dichroism (XMCD), and polarized neutron reflectometry (PNR) the possibility of magnetic axis rotation has been systematically studied and excluded. These results suggest that the Verwey transition takes place in the vicinity of a high-spin to low-spin transition of the Fe^{2+} ions, a phenomenon which is well known for $3d^6$ compounds, but has been neglected so far for Fe_3O_4 [20]. Existing Mössbauer experiments for single crystals so far have not produced evidence towards a spin state crossover [21]. Due to the low mass of ^{57}Fe in thin films, we were not able to do Mössbauer so far. Here, we argue that the huge change in magnetization at the Verwey transition is associated with a spin state crossover. We conclude that the Verwey transition occurs at the vicinity to a magnetic crossover instability, and, therefore, the degree of the crossover transition depends on sample synthesis.

Thin films of Fe_3O_4 were deposited on *c*-cut Al_2O_3 [*c*-Al_2O_3, i.e., α-Al_2O_3 (0001)] substrates using radio frequency-magnetron sputtering. Prior to deposition the sapphire substrates were heated and annealed for 10 min inside the sputtering unit at a temperature of 825 K in an oxygen environment of 10^{-5} mbar. Following this annealing step the chamber was pumped down to a pressure of 10^{-7} mbar, and the iron target was presputtered. During deposition of the 40 nm thick films, the substrate temperature was kept at 725 K in an oxygen flow of 0.2 sccm and an Ar flow of 4 sccm. After deposition the films were cooled at a rate of 25 K/min in a pure Ar atmosphere. The thin films were characterized by x-ray diffraction (XRD) and reflectometry (XRR) [using a Rigaku SmartLab rotating anode thin film diffractometer with Cu anode ($\lambda = 1.5406$ Å)] and by SQUID magnetometry (Quantum Design MPMS). PNR was performed at the D17

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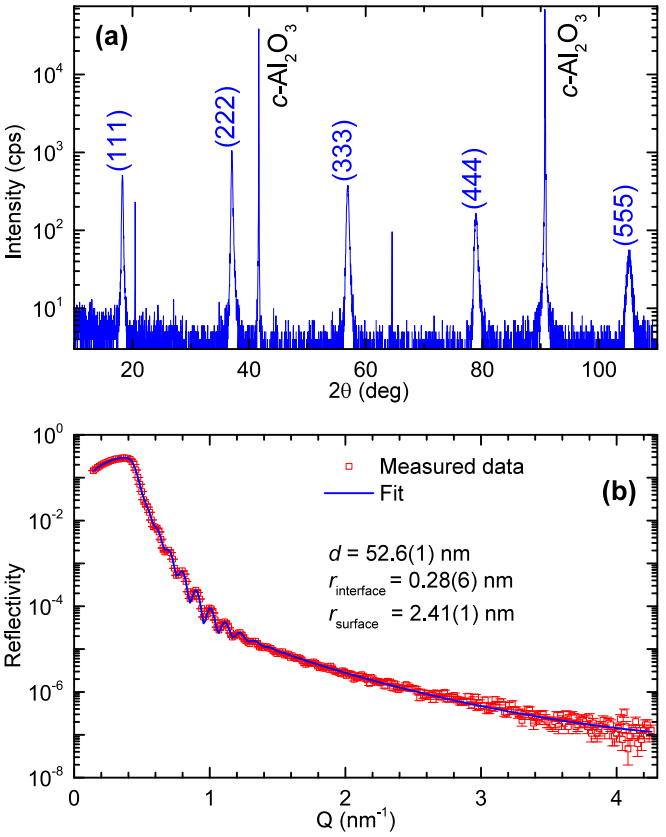

FIG. 1. (a) Out-of-plane x-ray ($\lambda = 1.5406$ Å) diffraction pattern of Fe_3O_4 on c-Al_2O_3. (b) X-ray reflectometry data of the same film. From the data refinement (using GenX [24]) the film thickness, and the interface respectively surface roughness were obtained.

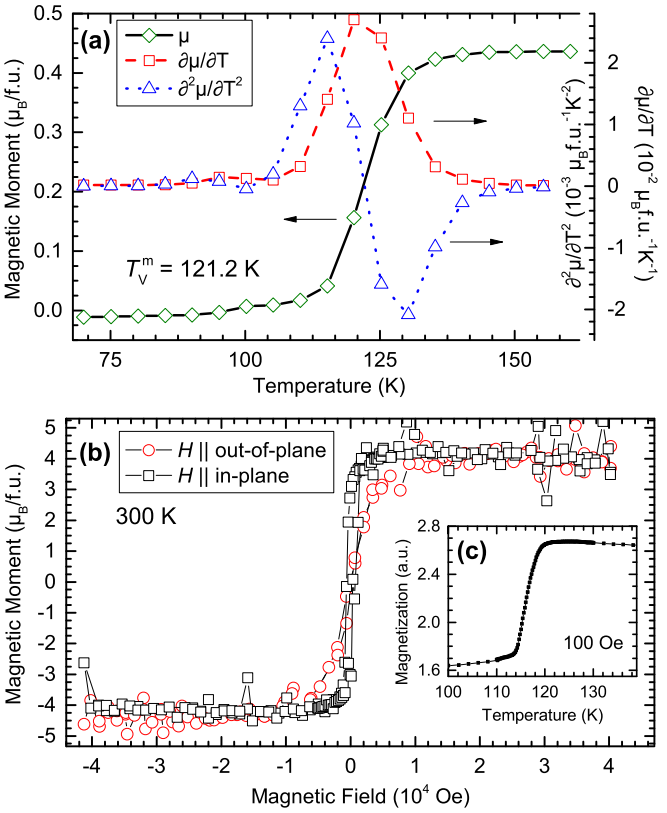

FIG. 2. (a) Magnetic moment vs temperature for the magnetite thin film on c-Al_2O_3. The first and second derivative in the vicinity of the transition are shown. (b) Magnetic hysteresis curves recorded for field applied in-plane and out-of-plane of the thin film. (c) Magnetic moment vs temperature for a magnetite single crystal.

beam line of the Institut Laue-Langevin (ILL), operated in the monochromatic, polarized beam mode. XMCD measurements at the Fe K-edge were preformed at the ID12 beamline of the European Synchrotron Radiation Facility (ESRF).

It is well known that small deviations in synthesis may lead to very different characteristics of the Verwey transition in magnetite samples [2,13,22]. We have performed this study using consistently one sample exhibiting a large change in magnetization across the Verwey transition. We have summarized the structural and magnetic characterization of this sample in Figs. 1 and 2. From the XRD pattern as shown in Fig. 1(a), the c-axis lattice constant was found to be $c = 8.397$ Å using the Nelson-Riley method. This value is very close to the single-crystalline value. The out-of-plane epitaxial relation between c-Al_2O_3 and Fe_3O_4 is given by Al_2O_3 (0001) ∥ Fe_3O_4 (111). The in-plane epitaxial relation is given by Al_2O_3 [$10\bar{1}0$] ∥ Fe_3O_4 [$11\bar{2}$], in agreement with previous work [23]. The thin film exhibited an out-of-plane mosaicity of 0.12° (full width of half maximum of the rocking curve). The XRR was best refined (using the program GenX [24]) by assuming an interface roughness of 2.8 Å to the substrate and a 2.4 nm surface roughness [see Fig. 1(b)].

SQUID magnetization measurements are shown in Figs. 2(a) and 2(b). The measurements were performed as follows: the sample was zero field cooled (ZFC) from room temperature to $T = 60$ K. Then, it was heated to $T = 160$ K in a field of 100 Oe applied parallel to the [$11\bar{2}$] direction of the film. We have determined the Verwey transition temperature as obtained from this magnetization, M, measurement, $T_V^M = 121.2$ K, from the maximum of the first derivative of the $M(T)$ curve. The peaks of the second derivative mark the onset and endpoint temperature of the Verwey transition. The difference in onset and endpoint temperature is defined as the transition width, $\Delta T \approx 15$ K. The drop in magnetization corresponds to a giant change of several 1000%. The sharp decrease of magnetization at the Verwey transition with decreasing temperature is common for magnetite films [25–27] and single crystals [see Fig. 2(c)]. The sharp change in magnetization is most prominent in low fields; with increasing field the magnetization change decreases [27]. The saturation magnetic moment, μ_S, of the sample is very close to $4\mu_B$/f.u. [$\mu_S = 3.85\mu_B$/f.u. (or 462 emu/cm^3) in the thin film as compared to $\mu_S = 3.78\mu_B$/f.u. of a commercially available single crystal]. Recently, even films with bulklike ∼480 emu/cm^3 have been reported [28]. From the analysis of the Raman data [12,29] and comparing it to standard bulk values [30], as well as the low saturation field, we conclude that the density of antiphase boundaries in the sample is low. The magnetization decrease seems to be a general trend in magnetite samples. So far, we have mostly mentioned thin film data, but also magnetite single crystals show a clear decrease of magnetization [15,16]. Applying exactly the same

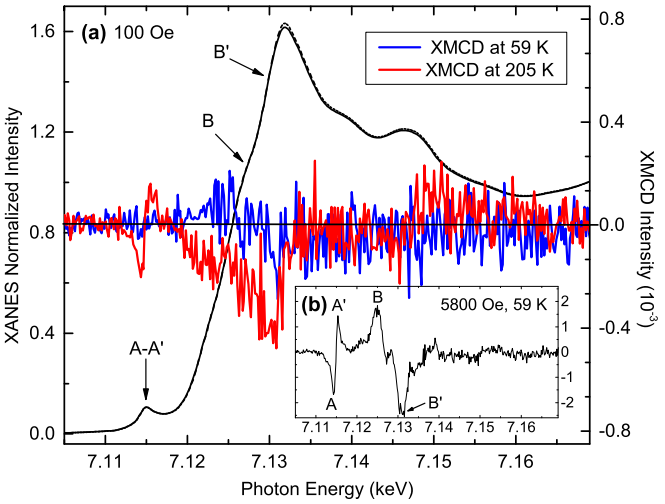

FIG. 3. (a) K-edge XANES and x-ray magnetic circular dichroism of a Fe_3O_4 thin film grown onto a c-cut Al_2O_3 substrate, measured in an applied field of 100 Oe. The arrows indicate the typical pre-edge features of magnetite. The resulting XMCD spectra are shown in dark gray (blue) in the Verwey state at $T = 59$ K and in light gray (red) for $T = 205$ K above the Verwey transition. (b) XANES of the same sample at $T = 59$ K, $H = 5.8$ kOe.

measurement ZFC SQUID procedure as we did for thin films, a commercial magnetite single crystal [see Fig. 2(c)] also shows a relatively large and sharp decrease of magnetization.

X-ray absorption near edge spectroscopy (XANES) and the XMCD spectra of magnetite have been previously studied for single crystals, powder, and thin film samples [31–34]. In these studies, there was no significant change in magnetic moment observed when the Verwey temperature is crossed [31,35]. We have performed XMCD measurements at the K edge of Fe as shown in Fig. 3. For the XMCD experiment the sample was mounted in the same way as in the SQUID with the $[11\bar{2}]$ direction parallel to the applied magnetic field. Also, the same ZFC procedure was applied. One spectrum was taken below the Verwey transition at 59 K and one above the Verwey transition at 205 K. XMCD at the K-edge does not allow the use of the well established magneto-optical sum rules, applicable to the $L_{2,3}$ or $M_{4,5}$ edges, from which the spin and orbital magnetic moments can be derived. The K-edge dichroism of Fe is a result of the spin and orbital polarization in the $4p$ valence shell [36]. The polarization of the $4p$ states predominantly originates from the on-site and intrasite exchange interactions with the polarized $3d$ states; therefore, changes in the $3d$ polarization will also affect the K-edge XMCD intensity [37]. One advantage of using the necessary high photon energies for K-edge experiments in Fe is the larger penetration depth yielding information of the whole thin film, in contrast to L-edge spectroscopy being restricted to a few nanometers at the sample surface. The K-edge XANES spectrum in Fig. 3(a) shows the typical pre-edge features $A - A'$ and $B - B'$ for Fe_3O_4 (marked with arrows). A clear change in the XMCD intensity was observed below the Verwey transition temperature, T_V. While there was a finite signal above the Verwey transition indicating the magnetization of the Fe ions, the signal almost fully disappeared at 59 K. The other important change in the XMCD signal for $T < T_V$ is the disappearance of the $A - A'$ feature at 7.115 keV observed above T_V in the XMCD and XANES spectra. This feature has been attributed predominantly to the tetrahedral Fe^{3+} sites [37,38]. The unusually broad B' and seemingly missing B peaks return to their sharper form known from literature [37] on the application of a large magnetic field [see Fig. 3(b)]. Even though there is no general theoretical interpretation available and the correlation between the crystallographic coordination and the energy spectrum is still unclear, it seems to be obvious that there is a drastic change in the magnetic polarization when passing into the Verwey state. In magnetite, a pressure dependent change in the XMCD signal has been reported [37]. As a possible interpretation, a transition from a high-spin to intermediate-spin state of the octahedral Fe^{2+} ions has been suggested. This experimental result has not been confirmed in a second measurement by another group [39]. Obviously, the spin state crossover depends on sample properties and actual measurement conditions. In [37] the applied field is not given, but comparison of the XMCD spectra to the high field result in Fig. 3(b) suggests that a relatively large field was applied, well above the stability of the intermediate or low spin state. In order to overcome those issues, we have performed the experiments described in this paper consistently on one well characterized sample. Furthermore, ^{57}Fe NMR studies have been interpreted previously involving a transition from an inverse spinel structure to a normal spinel structure below T_V [17,40]. As the $3d^6$ state of Fe^{2+} is prone to spin state transitions, these experiments indicate a possible change of the intrinsic magnetization in Fe_3O_4. The SQUID and XMCD experiments reported here further corroborate these results showing a clear change in magnetization of the magnetite thin film sample with almost vanishing magnetization in the Verwey state.

We have performed polarized neutron reflectometry in order to study the change in magnetization across the Verwey transition in more detail. PNR allows the simultaneous measurement of the magnetic moment profile and its orientational behavior due to spin rotation or noncollinearity. In Fig. 4 we show the reflection amplitudes R^{++}, R^{--}, and the spin-flip amplitudes R^{+-} for temperatures above and below T_V and for two fields (100 and 1000 Oe). R^{++} and R^{--} describe neutrons with parallel or antiparallel spin orientation with respect to the applied field that do not change their polarization state. The difference between R^{++} and R^{--} allows the determination of the in-plane magnetic moment. The amplitudes were corrected for polarization inefficiencies, beam divergence, and wavelength resolution [41,42]. R^{+-} and R^{-+} describe scattering processes involving a change of magnetization due to an in-plane contribution of the magnetization which is not collinear to the applied field. The reflection amplitudes R^{+-} and R^{-+} allow a rotation analysis [43]. The results of the PNR experiments are summarized in Fig. 4. First, the measurement conditions of the SQUID and XANES were reproduced in the PNR experiment [see Figs. 4(a) and 4(b)]. The sample was cooled in zero field below the Verwey transition temperature and an external field of 100 Oe was applied parallel to the $[11\bar{2}]$ in-plane direction of the film. The simultaneous fit of R^{++} and R^{--} gave $\mu = 0.11 \pm 0.07 \mu_B$/f.u. at 40 K. Furthermore, spin-flip amplitudes did not indicate any

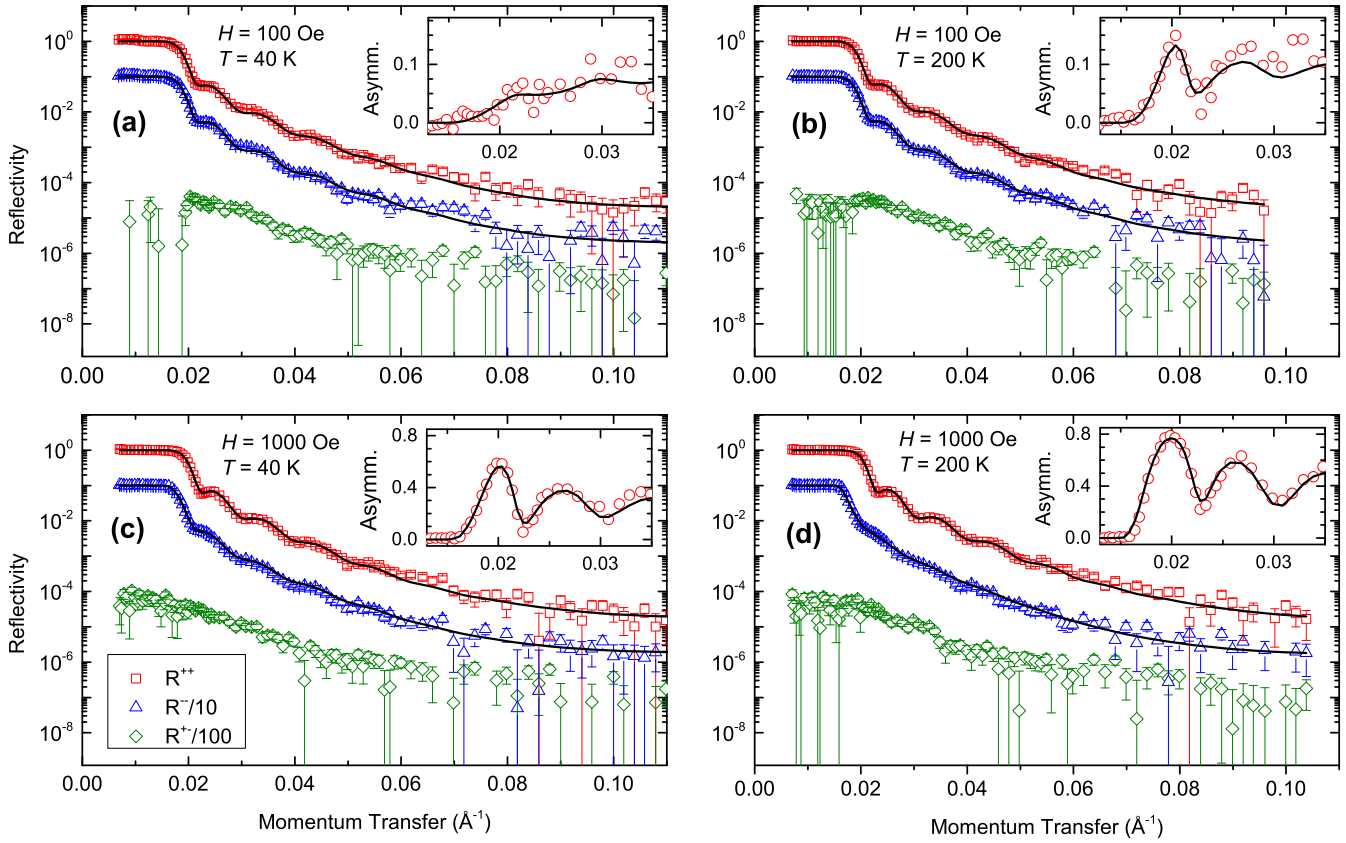

FIG. 4. Polarized neutron reflectivity of a magnetite thin film for different applied magnetic fields and temperatures. The data (open symbols) have been corrected for polarization efficiency. The lines are fits. The remanent spin-flip scattering, R^{+-}, is due to imperfect corrections. The insets show the spin asymmetry, $(R^{++} - R^{--})/(R^{++} + R^{--})$, at low momentum transfer, calculated from data (circle) and fit (line). Note that the curves are shifted vertically for clarity. R^{--} has been divided by 10, and R^{+-} by 100.

substantial noncollinearity resulting from spin rotation. Above T_V, the magnetic moment increases to $0.3 \pm 0.13 \mu_B$/f.u., which is comparable to the moment measured using the SQUID-magnetometer ($0.4 \mu_B$/f.u.). In Figs. 4(c) and 4(d) the measurement procedure was repeated at the higher external magnetic field of 1000 Oe. There was again no indication of spin-rotation observable from the spin-flip reflectivities. At 1000 Oe and 200 K the film has a saturation moment of 74.3% of the bulk fully magnetized single crystal. A considerable drop in moment is measured below the Verwey transition, the moment drops to $1.53 \pm 0.06 \mu_B$/f.u. which corresponds to a reduction by 48.5%, without any indication of spin rotation. The results are summarized in Table I.

TABLE I. Summary of the resulting average magnetic moments and corresponding measurement error from PNR. The refinement was performed using the program GenX [24].

T (K)	H (Oe)	μ (μ_B/f.u.)	Error (μ_B/f.u.)	Change (%)
200	100	0.30	±0.13	
40	100	0.11	±0.07	−63.3
200	1000	2.97	±0.14	
40	1000	1.53	±0.06	−48.5

The here described experiments strongly suggest that an *intrinsic* reduction of the magnetic moment is an ingredient of the Verwey transition. There is still the loophole that the magnetic moments in the Verwey state conspire in such a way, that all or a considerable part of about 50% of the in-plane oriented moments rotate by almost exactly 90° out-of-plane. This scenario is rather unlikely remembering the MFM experiment by Lee *et al.* reporting a tilting of the magnetic moment in magnetite from almost in-plane (86.5° with respect to the surface normal) to an angle of 80° with respect to the surface normal below the Verwey transition [19]. Although the low-spin (or intermediate-spin) state is rather unstable, since the population of high-spin states can be easily increased by applying a magnetic field, the experiments suggest that the Verwey transition takes place at the vicinity to a spin-state crossover. Here, we have presented evidence that the change in magnetization across the Verwey transition always involves at least a partial spin crossover. Spin-state transitions would, therefore, as in the case of the other magnetic $3d^6$ ion Co^{3+} [44,45], be part of the Verwey physics.

Starting from the observation of a giant change (>1000%) in magnetization as measured by SQUID of bulklike Fe_3O_4 thin films, we have applied consistently XANES/XMCD and PNR studies excluding a simple change of magnetic anisotropy in the samples. XMCD and PNR confirm a large negative change in magnetization across the Verwey transition.

The neutron reflectivity experiments give no evidence for a considerable rotation of the in-plane component of the magnetization vector. Therefore, the combination of the presented data suggests an intrinsic change of magnetic moment due a spin-state crossover into a fragile low or intermediate spin-state of Fe^{2+}. In summary, we suggest that the Verwey transition takes place in very close vicinity to a $3d^6$ spin-state transition.

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