Magnetic structure and phase transition at the surface region of Fe₃O₄(100)

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Abstract

The magnetic structure and phase transition of the near-surface region of Fe₃O₄(100) was investigated by ⁵⁷Fe conversion electron Mössbauer spectroscopy (CEMS) and theoretical calculations. It is revealed that at 300 K the magnetization is in-plane in the surface region and cants from the in-plane to the (111) direction in a deeper region suggesting the presence of a noncollinear magnetic structure. The critical exponents for the tetrahedral and octahedral sites are estimated to be 0.24 ± 0.01 and 0.28 ± 0.01, respectively. Near the critical temperature, furthermore, the magnetization direction in the surface region was found to deviate from the in-plane direction.

1. Introduction

Lowering of symmetry at surfaces exerts significant effects on the magnetic properties at surfaces such as spin orientation and phase transition, because the exchange interaction and magnetic anisotropy at surfaces are possibly modified as compared to the bulk. In the trend that the size of various devices is reduced to nanoscale, the magnetic properties at surfaces are increasingly important for device operation. Whereas surface magnetic anisotropy due to the broken symmetry could induce a noncollinear magnetic structure near surfaces [1, 2], modification of the surface exchange interaction might bring about critical phenomena characteristic to surfaces. It has been shown that the critical temperature and critical exponent can be different than those of bulk when the exchange interaction at the surface is different [3, 4].

Magnetite (Fe₃O₄) has attracted much attention due to its fascinating physical properties. It has an inverse-spinel structure consisting of two iron sites of tetrahedral A and octahedral B, where the A site is Fe³⁺ and the B site is a mixture of Fe³⁺ and Fe²⁺ at room temperature. Magnetite is a ferrimagnet, where spin 5/2 at the A site is antiferromagnetically coupled with spin 3/2 at the B site with a curie temperature of 858 K. The internal magnetic fields of the A and B sites are 48.9 and 45.2 T as measured by ⁵⁷Fe Mössbauer spectroscopy. The critical exponents of the magnetic phase transition are consistent with the value obtained by the three-dimensional Heisenberg model, although the values for the A and B sites are slightly different [5, 6].

Magnetite thin films with various thicknesses have been grown on substrates and their magnetic properties have been studied in detail. The magnetization direction is determined by the competition between the bulk magnetocrystalline anisotropy, surface/interface magnetocrystalline anisotropy, exchange interaction, and the shape anisotropy [7–9]. Due to the large shape anisotropy energy, the magnetization of thin films tend to be in-plane [10–12]. When the film thickness increases, it is reported that the magnetization direction changes from in-plane towards the bulk easy magnetization direction [13]. This might point to a non-collinear magnetic structure, although the magnetic structure was tacitly assumed to be collinear in the entire film. It is also reported that the magnetic anisotropy of the Fe₃O₄ films are controllable by the lattice distortion and electric coupling at the interface [13–16]. Although the magnetic phase transition at the surface of Fe₃O₄ is reported [17],
the difference of the magnetic critical behavior between the surface region and bulk of Fe₃O₄ is yet to be clarified. The surface magnetic anisotropy plays an important role for the magnetic properties of nanoparticles. Due to the surface effect, the shell part of nanoparticles could have a spin-canting structure as compared to the core part [18–20], which also corresponds to a noncollinear magnetic structure.

To clarify the surface magnetic properties, depth-resolved investigation of the magnetic properties is strongly required. The conversion electron Mössbauer spectroscopy (CEMS) probes the ⁵⁷Fe nuclear levels by detecting the internal conversion electrons from the sample. Since electrons suffer from energy loss when passing through solids, energy-resolved CEMS allows for depth-resolved analysis of the magnetism of materials [21]. Furthermore, by doping ⁵⁷Fe in thin films, ⁵⁷Fe CEMS can selectively probe the ⁵⁷Fe doping layer, which allows depth-selective investigation of magnetic properties [22, 23].

We recently revealed the presence of noncollinear magnetic structures at the surfaces of iron and magnetite [24, 25]. We also suggested that the magnetic phase transition at the interface is different from that in the film for an iron film epitaxially grown on MgO(100) [9]. The magnetic critical behavior at the surface region of magnetite may also be different from that of bulk, which is expected to give important information for the interpretation of the surface magnetism of the magnetite. In the present study, we investigated the magnetic structure and phase transition at the surface region of Fe₃O₄(100) by means of CEMS and theoretical calculations. It is revealed that the magnetization is in-plane in the surface region and cant from the in-plane to the (111) direction in a deeper region. It is also shown that the critical exponent at the surface region is significantly smaller than those for bulk magnetite suggesting that the surface region is described by the two-dimensional XY model rather than the three-dimensional Heisenberg model. As the temperature rises above 700 K, furthermore, the magnetization is observed to deviate from the in-plane direction.

2. Experimental and theoretical methods

The sample used in the present study is the (100) surface of a single crystal Fe₃O₄. To enhance the surface sensitivity of Mössbauer spectroscopy, we fabricated a ⁵⁷Fe₂O₃ layer by depositing ⁵⁷Fe (95% enriched) on the Fe₃O₄(100) surface with a thickness of 20 nm at a rate of 0.01 nm/s in oxygen of 3 × 10⁻⁴ Pa at 550 K after cleaning the surface in an ultra-high vacuum chamber (base pressure: 1 × 10⁻⁸ Pa) [25]. The ⁵⁷Fe₂O₃ was epitaxially grown as confirmed by reflection high energy electron diffraction.

The magnetic property of the sample was investigated by CEMS using a conventional ⁵⁷Co radioisotope with a γ-ray energy of 14.413 keV. The electron emitted via the internal conversion process associated with the deexcitation of ⁵⁷Fe nuclei was detected with a sealed-off proportional counter filled with rare gases. In the present study, a mixture of Ar (10%) and He (90%) was used, which improved the energy resolution to ∆E/E = 30% compared to ∆E/E = 80% of the pure-He-filled proportional counter. The internal magnetic field and magnetization direction were analyzed by the Zeeman-split sextet of the CEMS spectrum.

The CEMS measurement was performed in a depth-selective manner. Figure S1 available online at stacks.iop.org/JPCS/2020/115001/mmedia shows an output pulse height distribution of the proportional counter, which corresponds to the energy spectrum of the electrons emitted from the surface [26]. The main peak denoted by b corresponds to the K-shell conversion electron with an energy of 7.3 keV. The energy spectrum also reveals a low-energy tail below 5.4 keV, which corresponds to the electrons emitted from the surface.

The depth range of the inelastically scattered conversion electrons was investigated in detail by measuring CEMS for Fe films with various thicknesses [27, 28]. The depth ranges for the energy ranges of 4.9–7.3 and 3.3–5.7 keV are estimated to be 0–260 and 200–350 nm, respectively. On the other hand, the absorption length of the incident γ-ray is determined by the ⁵⁷Fe nuclear excitation and the photoelectron excitation. The cross section of the former process is 2.56 × 10⁻¹⁸ cm², which is about 400 times larger than the latter [29, 30]. We can then estimate the absorption fraction of the incident γ-ray in a specific depth region. In the case of the Fe₂O₃ sample with a ⁵⁷Fe₂O₃ layer (20 nm), 20% of the incident γ-ray is absorbed within a depth of 0–20 nm. In depth regions of 20–260 nm and 200–350 nm, on the other hand, we estimate 4.8 and 3% of the incident γ-ray to be absorbed. By combining the above two effects, i.e. the electron escape depth and the γ-ray absorption depth, it follows that the predominant probing depth of the energy-resolved CEMS is 0–20 and 200–350 nm when the electrons at energies of 4.9–7.3 and 3.3–5.7 keV are detected, respectively.

The magneto-crystalline anisotropy (MCA) energy was evaluated by first-principles density-functional theory (DFT) calculations using the Vienna ab initio simulation package (VASP) [31–33]. In the calculations, wavefunctions were expanded in a plane-wave basis set, and the behaviors of core electrons were described by the projector augmented wave potential [34, 35]. For the exchange and correlation energy, the spin-polarized generalized gradient approximation proposed by Perdew, Becke, and Ernzerhof was adopted [36].
The (100) surface of Fe₃O₄ was prepared with 17 atomic layers of Fe₃O₄ along the [100] direction and 1.5 nm vacuum layer. The length of the tetragonal unit cell of Fe₃O₄ (100) was fixed to $a^2$, where $a = 0.839$ nm is the lattice constant of bulk Fe₃O₄. The surface structures of Fe₃O₄ (100) were fully optimized by relaxing the atomic positions. The k-point was considered with $31 \times 31 \times 1$ in the first Brillouin zone of the Fe₃O₄ (100) super-cell.

3. Results and discussion

3.1. Magnetic structure

3.1.1. Experimental results

Figure 1 shows the energy-resolved CEMS spectra at room temperature, where electrons with energies above 4.9 keV and of 3.5–5.7 keV are acquired. Two sets of six lines representing the Fe A and B sites are clearly observed. The origin of the horizontal axis in Mössbauer spectra is shown with respect to the center of the spectrum for $\alpha$-Fe. The solid curves are fits with fit parameters of the internal magnetic fields ($B_{hf}$), the intensity ratio of the transitions of $|\Delta m| = \pm 1$ ($I_{\Delta m = \pm 1}$) and $\Delta m = 0$ ($I_{\Delta m = 0}$), where $m$ is the magnetic quantum number of the $^{57}$Fe nuclear spin, the isomer shift ($\delta$) and the line widths of the peaks at the Fe sites. Because the quadrupole splitting is negligibly small at 300 K, the quadrupole splitting is excluded from the fit parameters in figure 1.

Magnetite often suffers from Fe deficiency described as Fe₃₋ₓO₄, where $x$ is the deviation from stoichiometric magnetite Fe₃O₄. In a previous study, the relation between the deviation $x$ and the intensity ratio of the tetrahedral site ($P_A$) to the octahedral site ($P_B$) is reported to be described as $P_B / P_A = (2 - 6x)/(1 + 5x)$. The ratio $P_B / P_A$ is clearly different in figure 1(a) and (b), where $x$ is estimated at 0.03 and 0.02, respectively. This indicates that Fe is slightly more deficient near the surface. The values of $\delta$ for the A and B sites are estimated at 0.277 ± 0.003 and 0.659 ± 0.003 mm s⁻¹, respectively, for both figure 1(a) and (b), which are similar to the
bulk values. The temperature dependences of \( \delta \) are also found to be similar to those of bulk [5]. This indicates that the chemical state of Fe atoms at the surface is similar to that of bulk.

The linewidth of the A (B) site spectrum is estimated at 0.24–0.28 (0.34–0.39) mm s\(^{-1}\) below 700 K, which is slightly larger than that of bulk [5, 38]. With increasing temperature, the width is broadened to 0.50 ± 0.07 (0.68 ± 0.07) mm s\(^{-1}\). Although the line broadening suggests enhanced diffusion of iron atoms [39], it is difficult to extract the diffusion coefficient from the line width because of other factors such as homogeneous and inhomogeneous broadening.

The magnetization direction of the sample can be evaluated from the intensity ratio of \( I_{\Delta m=\pm 1} \) and \( I_{\Delta m=0} \). The magnetization direction \( \theta_m \), as measured from the \( \gamma \)-ray direction is related to the intensity ratio as follows [40]

\[
I_{\Delta m=\pm 1}/I_{\Delta m=0} = (1 + \cos^2 \theta_m)/\sin^2 \theta_m
\]

In figure 1(a), the \( I_{\Delta m=\pm 1}/I_{\Delta m=0} \) values at the A and B sites are evaluated to be 1.01 and 1.07, respectively. This means that the magnetization direction of Fe\(_3\)O\(_4\) (100) is almost in-plane in the surface region within a depth of 20 nm. On the other hand, the magnetization directions of the A and B sites of figure 1(b) are estimated at 68 ± 2° and 66 ± 2° from the \( I_{\Delta m=\pm 1}/I_{\Delta m=0} \) values of 1.31 and 1.57, respectively. It is known that the easy, intermediate and hard axes of magnetization for magnetite are [111], [110] and [100] directions, respectively [41]. Since the experimentally obtained \( \theta_m \) values are larger than 55° corresponding to the bulk easy magnetization axis of (111), it is considered that the magnetization even in a deeper region of 200–350 nm is inclined to in-plane from the bulk easy magnetization direction. Note that the \( I_{\Delta m=\pm 1}/I_{\Delta m=0} \) value is slightly different between the A and B sites. This indicates the spin directions at the A and B sites are slightly different.

### 3.1.2. Theoretical calculations

The MCA energy of Fe\(_3\)O\(_4\) (100) was calculated by DFT using the force theorem [42]. The Fe\(_3\)O\(_4\) (100) surface is found to prefer in-plane magnetization with an MCA energy of \( K_{\text{sur}} = -1.75 \) erg cm\(^{-2}\). Note that the difference between the energy for the in-plane [001] and in-plane [011] is as small as 0.046 erg cm\(^{-2}\). A previous experimental study has shown that the easy magnetization direction of an Fe\(_3\)O\(_4\) (100) film is [011][43], which is consistent with the present results. The MCA energy for bulk Fe\(_3\)O\(_4\) on the other hand, is calculated as \( K_{\text{bulk}} = 1.09 \times 10^5 \) erg cm\(^{-3}\).

The origin of the in-plane MCA of Fe\(_3\)O\(_4\) (100) is analyzed by the second-order perturbation of the spin-orbit interaction using the wavefunctions obtained by DFT calculations on the basis of the formulation presented in [44]. With the spin–orbit coupling constant of 54 meV for Fe and 24 meV for O, the MCA energy in the second-order perturbation calculations is found to be consistent with the MCA energy from the first-principles calculations. Figure 2(b) shows the MCA energy at each atomic site of Fe\(_3\)O\(_4\) (100) denoted in figure 2(a), where the four spin–transition components of spin up-up, down-down, up-down and down-up are separately shown and the total contribution is denoted by the black bar. As seen in figure 2(b), the Fe B at the third atomic layer (Fe\(^{\text{III}}\)) predominantly contributes to the negative MCA energy (in-plane MCA) of Fe\(_3\)O\(_4\) (100). Interestingly, the Fe B at the surface (Fe\(^{\text{I}}\)) has a small contribution to the MCA energy because of the cancelation between the non-spin-flip terms (spin up-up and spin down-down) and the spin-flip terms (spin up-down and spin down-up). The Fe A at the sub-surface (Fe\(^{\text{II}}\)) also shows a small contribution to the in-plane MCA.

Clarifying the surface MCA energy, the stability of the noncollinear magnetic structure shown in figure 3(a) was evaluated by the following equation for the total energy (\( E_{\text{NC}} \))[9]:

\[
E_{\text{NC}} = K_{\text{sur}} \sin^2 \theta_{\text{sur}} + \int_0^W \left[ A \left( \frac{d\theta}{dz} \right)^2 + 2\pi M^2 \sin^2 \theta - K_{\text{bulk}} \left( \cos^2 \theta - \theta_{\{111\}} - \cos^2 \theta_{\{111\}} \right) \right] dz,
\]

where \( A = 2.64 \times 10^{-6} \) erg/cm [45] is the exchange stiffness constant and \( 2\pi M^2 \) is the shape magnetic anisotropy energy. Since the present experimental result shows the magnetization is in-plane within 20 nm from the surface, we assume the surface region as \( \theta_{\text{sur}} = 20 \) nm and \( \theta_{\text{sur}} = 0 \). We also assume that the angle of the spin direction (\( \theta \)) with respect to the surface in-plane (x axis) changes as \( \theta(z) = \theta_{\{111\}} \sin^2 (\pi (z - z_{\text{sur}})/2W) \) for \( z > z_{\text{sur}} \) (small changes from \( z \leq z_{\text{sur}} \), where the \( \theta_{\{111\}} = 35.3^\circ \) is the angle between the [111] and [001](x axis) directions, and \( W \) is the length of the tilted magnetic structure as shown in figure 3(a). When \( 2\pi M^2 = 1.58 \times 10^6 \) erg cm\(^{-3}\) as obtained by DFT calculations, \( E_{\text{NC}} \) is found to be positive irrespective of \( W \) indicating that the noncollinear magnetic structure is not stable. A previous study, on the other hand, has shown that the closure domain in magnetite is formed to reduce the magnetostatic energy [46] and the magnetostatic energy is reduced to 1/16 compared to a single magnetic domain [47]. Substituting \( 1.0 \times 10^6 \) erg cm\(^{-3}\) for \( 2\pi M^2 \), \( E_{\text{NC}} \) is plotted in figure 3(b) as a function of \( W \). It is found that the noncollinear magnetic structure is stabilized for \( W \gtrsim 200 \) nm.

From these results, it is concluded that the surface region of Fe\(_3\)O\(_4\) (100) has a noncollinear magnetic structure, where the magnetization direction changes from in-plane to (111) towards a deeper region with a
depth scale of ca. 200 nm. The in-plane magnetization observed on the (100) surface is similar to a previous study on the (111) surface. As discussed in previous papers, the noncollinear magnetic structure originates from the competition between the bulk magneto-crystalline anisotropy, surface magneto-crystalline anisotropy and magneto-static energy [9].

3.2. Phase transition and phonon
Figure 4 shows the CEMS spectra at various temperatures, where all electrons are acquired without energy resolution. Note that the CEMS spectra predominantly reflect the surface region of 0–20 nm because a $^{57}$Fe$_3$O$_4$ (20 nm) layer was formed on the sample surface. The Zeeman splitting is clearly decreased as the temperature increases from 578 to 862 K indicating a magnetic phase transition to a paramagnetic phase. An additional
paramagnetic component with a quadrupole splitting is observed at temperatures higher than 752 K. This additional component might be assigned to Fe$_{1-x}$O resulting from formation of oxygen vacancies at high temperature [48].

The temperature ($T$) dependences of the internal magnetic fields at the A and B sites are shown in figure 5 with fits by the following formula:

$$B_{hy}(T) = B_0 (1 - T/T_c)^\beta,$$

where $B_0$, $\beta$ and $T_c$ are fit parameters corresponding to the internal magnetic field at $T = 0$, the critical exponent and the critical temperature, respectively. The critical temperature is estimated at 861 K, which is consistent with a typical bulk value of ~858 K within the data scatter reported in literatures. Therefore, it is considered that $T_c$ at the surface is the same as the bulk value. The critical exponents of the A and B sites are estimated at 0.24 \pm 0.01.

Figure 4. CEMS spectra of $^{57}$Fe$_3$O$_4$(20 nm)/Fe$_3$O$_4$(100) at temperatures of (a) 578, (b) 752, (c) 814, (d) 845, and (e) 862 K. The solid curve in the middle of each figure is a fit of the entire spectrum, the components of the A site, B site and an additional site are shown below.
and 0.28 ± 0.01, respectively, which are significantly smaller than the values of 0.34 ± 0.01 at the A site and 0.32 ± 0.01 at the B site in the bulk [5]. The critical behavior near the Curie temperature is clearly different from those of bulk [5] as shown in figure 5. It is noted that the critical exponent of the B site is larger than that of the A site, which is also observed for bulk magnetite [5].

As theoretically investigated in previous studies [4], the surface critical exponent is lowered when the exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction. Although the in-plane exchange interaction is not evaluated in the present calculations, the interlayer exchange interaction at the surface is enhanced as compared to the bulk exchange interaction.

We next discuss the magnetization direction near the critical temperature. Figure 6 shows the $I_{\Delta m=\pm 1}/I_{\Delta m=0}$ value measured at various temperatures. It is almost 1.0 up to 725 K indicating that the magnetization directions of the A and B sites are in-plane. Above 725 K, on the other hand, the values seem to deviate from 1.0 and become larger. The larger $I_{\Delta m=\pm 1}/I_{\Delta m=0}$ value indicates either a magnetization direction of near-[111] or random orientation. Note that the $I_{\Delta m=\pm 1}/I_{\Delta m=0}$ value is confirmed to return to 1.0 when the sample temperature is lowered to room temperature from $T_C$. It has been shown that the magnetic anisotropy constant tends to get smaller with increasing temperature. We therefore argue here that the magnetization direction in the in-plane direction at room temperature becomes randomly oriented near the critical temperature.

The recoilless fraction ($f$) at a temperature of $T$ is expressed with the Debye temperature ($\Theta$) as [52]

$$f = \exp(-6E_rT/k\Theta^2) \text{ for } T > \Theta/2.$$  

Since $f$ is proportional to the total intensity of the CEMS spectrum, the Debye temperature can be estimated from the temperature dependence of CEMS. Because the total intensity $I$ of the six lines with respect to the background intensity ($I_{BG}$) is proportional to $f$, the temperature dependence of $I/I_{BG}$ is plotted as shown in figure 7. To avoid the effect of suspected oxygen vacancies above 750 K, equation (4) is fitted to the data in the range of 375–750 K. The Debye temperature ($\Theta_{A(B)}$) of the A (B) site is estimated at 191 ± 7 (194 ± 5) K, which

![Figure 5](https://example.com/f5.png)

**Figure 5.** Temperature dependence of the internal magnetic fields at the A (■) and B (○) sites. Solid curves are fits with equation (3). The error bars in the figure are evaluated by the statistical uncertainty of the CEMS spectra. The dash-dotted curves are the data taken from a previous study [5], which is scaled so that the Curie temperature matches the present data.
is lower than the bulk value of 334 (314) K \cite{52}. It is considered that the phonon in the surface region is softer than that in the bulk.

4. Conclusion

The magnetic structure and critical behavior in the surface region of Fe$_3$O$_4$(100) were investigated by CEMS and theoretical calculations. It is experimentally revealed that the surface region of Fe$_3$O$_4$(100) has a noncollinear magnetic structure in the depth direction, where the magnetization direction changes from in-plane to $\langle 111 \rangle$. The magneto-crystalline anisotropy energy at the Fe$_3$O$_4$(100) surface was estimated to be $-1.75$ erg cm$^{-2}$ by DFT calculations indicating preference of the in-plane magnetization. By evaluating the total magnetic energy taking account of the effects of the closure domain structure, the non-collinear magnetic structure was found to be stable when the noncollinear region is larger than 200 nm, which is in good agreement with the present
The critical temperature was similar to that of the bulk, the critical exponents were evaluated to be $0.24 \pm 0.01$ and $0.28 \pm 0.01$ for the A and B sites, respectively. Near the critical temperature, the magnetization direction at the surface seems to change from the in-plane direction to random orientation.

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