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Scanning Tunneling Microscopy Study of Surface Structure and Magnetism of Fe Thin Films Grown on MgO (001)

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Substrate preparation procedure dependence of the growth morphology and magnetic properties of 25 ML bcc-Fe(001) thin films epitaxially grown on MgO(001) substrates in a wide range of growth temperature was investigated by means of scanning tunneling microscopy (STM) and superconducting quantum interference device (SQUID). The growth morphology of Fe thin films was uniform both on a polished and on an annealed substrate, but nonuniform on a cleaved substrate. It was very difficult to obtain a flat Fe thin film on the cleaved substrate, and the film became discontinuous at or above a growth temperature of 493 K. At a growth temperature of 550 K, atomically defined terraces of Fe thin films were formed on the annealed substrate but were not formed on the polished substrate. A continuous film grown on the annealed substrate at a temperature of 593 K has a less magnetic anisotropy. The other continuous films have low coercivity of about 8 Oe and a biaxial magnetic anisotropy. The dependency of the growth morphology and magnetic properties of Fe thin films upon substrate preparation procedures concerning the presence of step-terraces on the substrate surface is discussed.

KEYWORDS: bcc-Fe(001), MgO(001), epitaxial growth, polished, annealed, cleaved, STM, SQUID, biaxial magnetic anisotropy

1. Introduction

An application of scanning tunneling microscopy (STM) to detect the spin-polarized surface states of a magnetic sample provides an exciting prospect of realizing a technique for imaging magnetic properties on the atomic scale. To demonstrate this possibility, a flat sample with a highly spin-polarized surface states is required. Since an Fe(001) surface shows a highly spin-polarized surface state,[1] this surface layer is expected to be suitable as a test sample for spin-polarized tunneling experiments.

In order to prepare a high-quality Fe(001) magnetic sample, we investigated epitaxial growth of Fe thin films on magnesium oxide (MgO)(001) substrates. MgO has received a considerable amount of attention as a substrate material for epitaxial growth of several metals, because it has a high melting point and can be easily obtained in large pieces. The reasons for choosing MgO as a substrate for preparing the Fe(001) surface are: (1) the lattice mismatch between bcc Fe ($a_{\text{Fe-bulk}} = 2.866$ Å) lattice and fcc MgO ($a_{\text{MgO}} = 4.213$ Å) sublattice is only 3.8%, and (2) an Fe monolayer on MgO(001) is predicted to have a high magnetic moment.[2]

In growth by molecular beam epitaxy (MBE), surface diffusion is the predominant mechanism for the transport of material in ordering processes. Preparing a substrate with an atomically flat step-terrace surface provides many advantages for producing high-quality overlayer properties, because the monolayer height steps lead the atoms for diffusion along step edges. However, it is difficult to obtain such a surface on MgO(001) substrates. A cleaving procedure is the simplest way to obtain well-ordered surfaces with step-terrace structures, but the step height and the terrace width have been found to be nonuniform.[3,4] Mechanically polished substrates exhibit a flat surface, but defined stepped surfaces could not be obtained because the surfaces were damaged by the polishing procedure.[5,6] High-temperature annealing in oxygen of the mechanically polished substrate improved the surface, resulting in well-defined steps with step height in the range of a unit cell height (0.421 nm) to 5 nanometers.[6–10] There are a few reports on uniform monolayer height (0.21 nm) steps on substrates.[5,10] However, annealing above 1123 K leads to the segregation of impurities, commonly calcium, to the MgO surface.[6–8,11,12] This segregated Ca has been found to degrade the microstructures and superconductivity of DyBaCuO films.[12] Furthermore, annealing for a long period of time also degrades the uniformity of growth morphology and increases the rms roughness of the Fe thin films.[10]

We report on an investigation of growth morphology and magnetic properties of Fe thin films epitaxially grown on polished, annealed and cleaved MgO (001) substrates at a wide range of growth temperatures (T_g). We succeeded in preparing well-ordered, atomically flat MgO surfaces by employing an annealing for a short period of time on polished substrates. This annealed substrate is suitable for improving the Fe thin film properties. In this paper, the effects of the presence of step-terraces on the surface of the substrate, which have not been discussed in previous works,[9,13–21] is investigated. We will discuss the growth morphology and magnetic properties of the Fe thin films on an annealed substrate with uniform monolayer height steps and on a cleaved substrate with nonuniform height steps. The growth morphology was characterized by scanning tunneling microscopy (STM), and the magnetic properties were explored by superconducting quantum interference device (SQUID) measurements.

2. Experiment

Commercial mechanically polished single crystal MgO (001) substrates were divided into two batches for different preparation procedures. One batch was ultrasonically cleaned in acetone, isopropanol and methanol, then exposed to boiled acetone at 328 K and finally rinsed in isopropanol and methanol. The other batch was annealed in oxygen environment at 1273 K for 30 min. after cleaning by the same procedures described above. The annealing was performed

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in a period of time as short as possible to suppress the segregation of Ca to the MgO surface. Furthermore, we prepared other substrates by cleaving in air.

Narrow Au electrodes were deposited on two edges of a MgO substrate, and the substrate was mounted on a tantalum sample plate by mechanical clipping. Then the substrate was immediately introduced into the ultra high vacuum (UHV) system. The substrate was flashed up to 1100 K over three times to remove carbon contamination. The surface structure and morphology of MgO were evaluated by atomic force microscopy (AFM) and reflection high energy electron diffraction (RHEED).

Fe films were grown at a deposition rate of 0.5 monolayer (ML; 1 ML $=$ 0.143 nm) min. up to 25 ML in thickness. Deposition was done at a growth temperature ranging from 300 K and 633 K. The pressure during evaporation never exceeded 3.0×10^{-8} Pa. The surface structures and growth morphology of the Fe films were characterized by RHEED and STM.

The magnetic properties were investigated by SQUID measurement. Prior to SQUID measurement, a 3 nm thick Au film was evaporated on the Fe thin film at room temperature in the UHV system to prevent surface oxidation. SQUID measurement was carried out at 300 K. A magnetic field was applied in the plane of the sample surface.

3. Results and Discussion

3.1 Surface morphology of substrate

Table I shows a summary of substrate preparation procedures and surface morphology. The surface morphology was determined by AFM in UHV before Fe deposition. The polished substrate exhibits a flat surface with average rms surface roughness of about 0.15 ± 0.03 nm. Although the polished substrates exhibited a flat surface feature, we did not observe a defined step-terrace surface structure.

Due to the annealing procedure on the mechanically polished substrate, the surface changed drastically into a step-terrace structure surface, as shown in Fig. 1. This annealed substrate exhibits a well-ordered surface with about 100 nm to 200 nm wide terraces separated by one monolayer height steps. The steps run along the ⟨011⟩ direction over 4 microns.

The cleaving procedure produced a step-terrace surface structure with nonuniform terrace width and step height. This cleaved substrate exhibits 10 nm to 400 nm wide terraces that are separated by straight steps running along the ⟨001⟩ direction over 5 microns. The steps mostly ranged between 0.21 nm and 1 nm in height, however high steps of about 5 nm to 10 nm were also observed. Some rough regions that show degradation of step-terrace features were also observed. These must be caused by chemical interaction with atmospheric water vapor during preparation.[3)]

3.2 Growth morphology

3.2.1 RHEED investigation

RHEED patterns revealed that all Fe thin films grown on each substrate showed a similar epitaxial relation between Fe thin films and MgO(001) substrates. The patterns indicated an epitaxial growth of bcc-Fe(001) on MgO(001) with an epitaxial relation of [110](001)Fe//[100](001)MgO.

RHEED patterns also indicated an evolution of surface roughness of the Fe thin films with the increase of growth temperature. The patterns became streaky as the growth temperature was increased up to 550 K. However, the patterns became spotty again when the growth temperature was increased from 550 K up to 633 K. These evolution patterns indicate that the surface structure of the Fe thin films strongly depends on the growth temperature. The surface roughness of the Fe thin films decreased with increasing growth temperature up to 550 K, but became rough with increasing growth temperature to 633 K. This tendency did not depend on the substrate preparation procedures.

3.2.2 Growth morphology on the polished substrate

Figure 2 shows STM images of the surface morphology of Fe thin film grown on polished substrates. All images have a scan size of $50 \times 25\,\mathrm{nm}^2$. The images show that growth morphology evolution depends on the growth temperature. In the growth temperature range from 300 K to 550 K, the island size

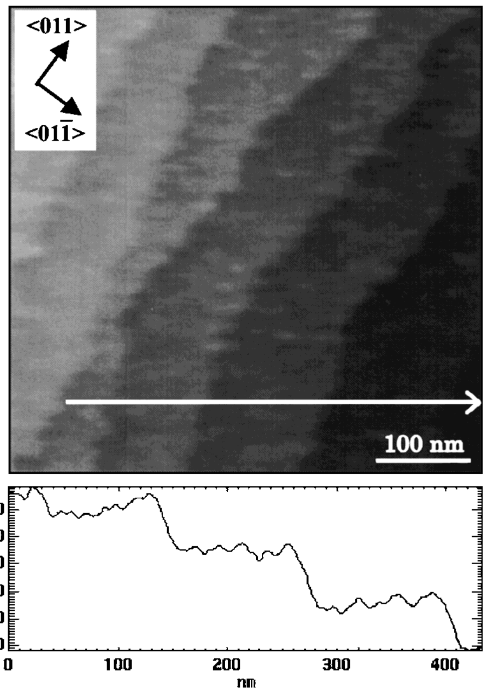

Fig. 1. AFM image of a well-ordered annealed MgO (001) surface. Image scan size is $500 \times 500\,\mathrm{nm}^2$. Most of the terraces range between 100 to 200 nm in width and are separated by monolayer height steps. The steps run along the ⟨011⟩ direction.

Table I. Summary of substrate preparation procedures and surface morphology.

Substrate	Preparation procedures	Surface structure
polished	mechanically polished	does not exhibit defined step-terrace structure
annealed	annealing of mechanically polished	exhibits defined step-terrace structure with uniform monolayer height steps
cleaved	cleaving in air	exhibits defined step-terrace structure with nonuniform step height and terrace width

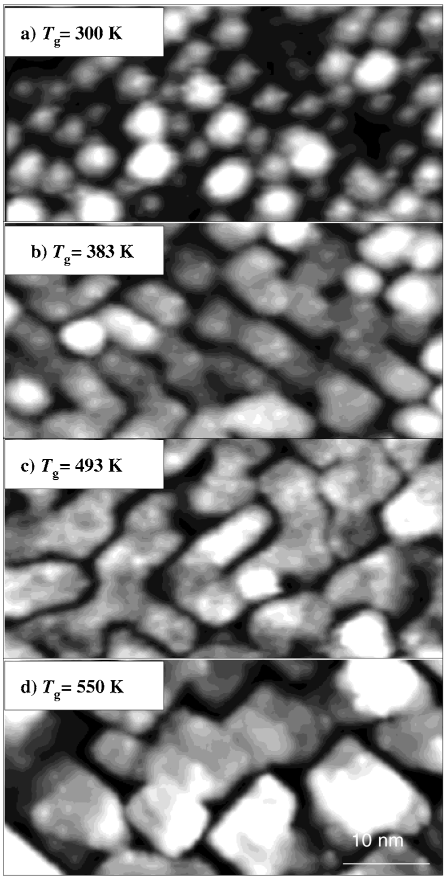

Fig. 2. STM image of 25 ML Fe thin films epitaxially grown on polished MgO (001) substrates for growth at (a) 300 K, (b) 383 K, (c) 493 K and (d) 550 K. Scan size of all images is 50 × 25 nm^2. The average island size is (a) 4.82 nm, (b) 5.37 nm, (c) 6.68 nm and (d) 10.05 nm, respectively. The rms roughness is (a) 0.28 nm, (b) 0.26 nm, (c) 0.25 nm and (d) 0.23 nm, respectively.

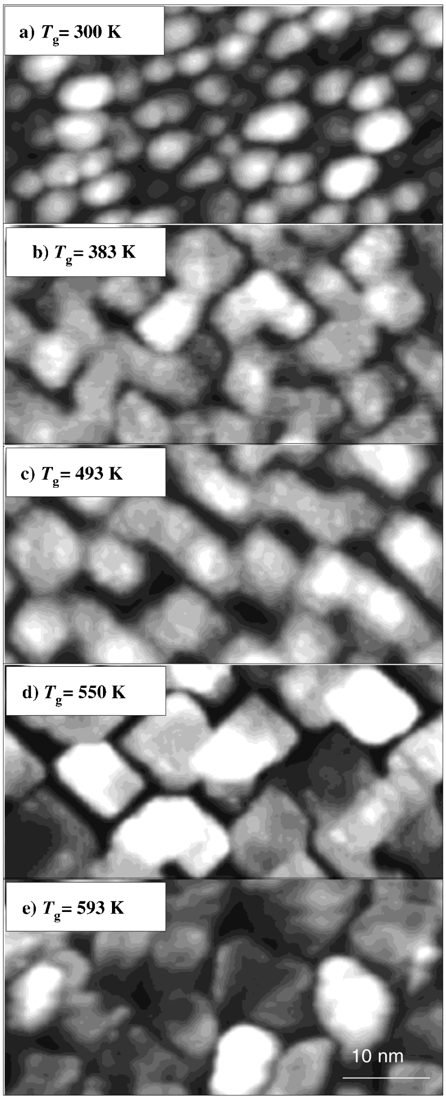

Fig. 3. STM images of 25 ML Fe thin films epitaxially grown on annealed MgO (001) substrates for growth at (a) 300 K, (b) 383 K, (c) 493 K, (d) 550 K and (e) 593 K. Scan size of all images is 50 × 25 nm^2. Average island size is (a) 4.90 nm, (b) 5.47 nm, (c) 6.70 nm, (d) 10.02 nm and (e) 8.56 nm, respectively. The rms roughness is (a) 0.26 nm, (b) 0.25 nm, (c) 0.23 nm, (d) 0.22 nm and (e) 0.23 nm, respectively.

increased, the rms roughness decreased and the island shape became squarer and formed a flat surface, as growth temperature increased, as shown in Figs. 2(a) to 2(d). The Fe thin films became discontinuous at or above a growth temperature of 593 K.

The Fe thin film grown at 300 K, as shown in Fig. 2(a), shows round islands. This round shape becomes square as the growth temperature is increased to 383 K, as shown in Fig. 2(b). The island edges are aligned along the Fe[100] direction. Streaky RHEED patterns appeared at this growth temperature, 383 K, revealing formation of a flat surface.

Increasing the growth temperature from 383 K to 493 K increased the island size and decreased the rms roughness of the Fe thin film, as shown in Fig. 2(c). As shown in Fig. 2(d), the flat terraces are formed with increasing growth temperature from 493 K to 550 K. The steps, if observed, were found to be of monolayer height and aligned along the Fe[100] direction. In these terraces, small rounded islands are observed within the terraces.

A decrease of rms surface roughness, increase in island size, and formation of square shape islands and terraces are consequences of the increased mobility of the atoms to diffuse along step edges. This growth morphology evolution depends on the growth temperature, which is consistent with previous studies.[9,13)]

Jordan *et al*. reported that 595 K is the optimum temperature to produce flat terraces of 5 nm thick Fe thin film.[13)] We found that increasing the growth temperature to 593 K causes formation of discontinuous films. This is most likely caused by the difference of the film thickness. In our work, the film was only 25 ML (about 3.58 nm), which is thinner than their film of 5 nm. thick.

3.2.3 Growth morphology on the annealed substrate

Figure 3 shows the STM images of Fe thin films grown on annealed substrates. The images reveal that the dependency of growth morphology evolution of Fe thin films on growth temperature is similar to that of films on polished substrates. However, the island size was typically bigger and the rms surface roughness was smaller than the films grown on the polished substrates.

The effects of the substrate annealing procedure on the growth morphology of Fe thin films were marked for the Fe thin films grown at or above a growth temperature of 493 K. At a growth temperature of 493 K, steps appeared on the islands. At 550 K, terraces with a more clearly defined squarer shape and defined straight steps were formed. The width of terraces typically ranged between 5 nm and 10 nm. At a growth temperature of 593 K, the Fe thin film was continuous.

These advantage points revealed that substrates annealed for a short period of time available to improve the surface properties of Fe thin films more markedly than polished substrates. Considering the presence of uniform monolayer height steps on the annealed substrates, these steps should play an important role of improving the mobility of the atoms to diffuse along step edges at the first monolayer films, which then leads to formation of flat terraces films.

The Fe thin film grown at the maximum growth temperature at which continuous films are produced, 593 K, formed rough, disordered and rounded islands, as shown in Fig. 3(e). This is inconsistent with a previous study that reported formation of flat terrace films.[13] Since the higher temperature, 633 K, produced a discontinuous film, we concluded that a growth temperature of 593 K is the optimum temperature for producing continuous films but is not the optimum temperature for producing flat terraces films. We predicted that 593 K is a critical growth temperature at which the flat terrace structures started to break into disordered, rough islands. These islands then formed discontinuous film at higher temperatures.

3.2.4 Growth morphology on the cleaved substrate

The growth morphology of Fe thin film grown on a cleaved substrate at 300 K was found to be similar with films on both the polished substrate and on the annealed substrate. However, films grown at a higher temperature, 383 K, showed a different morphology.

Figures 4(a) to 4(d) shows topographic STM images of an Fe thin film grown on the cleaved substrate at 383 K. Each image was taken at a different region on the same sample and had a scan size of $100 \times 50\,\mathrm{nm}^2$. The images reveal that the cleaved substrate resulted in nonuniform growth morphology of the Fe thin film. Due to the cleaving procedure that produced the nonuniform step-terrace surface, formation with different growth morphology, which depends on the substrate structures were observed.

Figure 4(a) shows a region with the same morphology as films grown on the polished substrate, as shown in Fig. 2(b). The morphology is the same as that of the film grown on the annealed substrate, as shown in Fig. 3(b). Square pyramidal islands were formed. This kind of island formation was observed on the large terraces lying near the high steps of about 5 nm to 10 nm.

Figure 4(b) shows formation of islands of uniform size that ordered in the Fe[100] direction, similar to Fig. 4(a). However, the islands are small and round, which results in a rougher surface. Since a partially ordered island structure exists, it should be expected that the film in this region would grow on clean, defined terraces, however, the terraces were narrow and separated by steps higher than monolayer height.

Figure 4(c) shows the roughest surface region, which is formed by disordered and nonuniform round islands. One of the reasons why the disordered structure is formed is the degradation of substrate surfaces, which is caused by chemical interaction with atmospheric water vapor during preparation.

On the other hand, a region that shows very flat terraces with defined steps was also observed, as shown in Fig. 4(d).

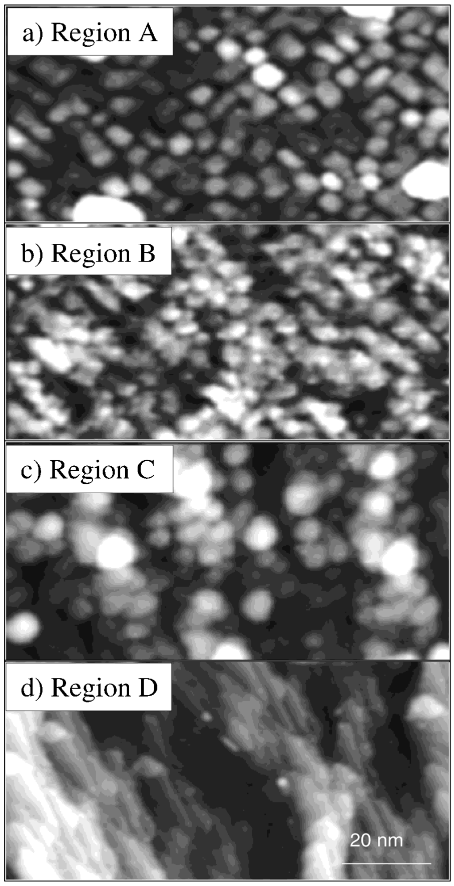

Fig. 4. STM images of a 25 ML Fe thin film epitaxially grown on a cleaved MgO (001) substrate for growth at 383 K. Each image was taken at a different region on the same sample. Scan size of all images is $100 \times 50\,\mathrm{nm}^2$. The rms roughness is (a) 0.25 nm, (b) 0.27 nm, (c) 0.33 nm and (d) 0.21 nm, respectively.

The steps have monolayer height and are mostly aligned along in the Fe[100] direction within a 15% error. However, steps aligned along the Fe[110] direction parallel to the cleaved MgO steps direction were also observed.

The STM image of a film grown at a higher temperature of 493 K could not be obtained because the film becomes discontinuous. Considering that this temperature is sufficiently high, that is, higher than the critical temperature where the Schwoebel barrier is overcome,[9] at this temperature the Fe atoms can diffuse not only in an upward but also in a downward direction. Thus, we concluded that high steps of about 5 nm to 10 nm on cleaved surfaces broke the continuity of the film that is only 3.58 nm thick.

3.2.5 Discussion

Thurmer *et al.* observed atomically flat terraces of Fe films grown at a growth temperature above 500 K.[9] Jordan *et al.* reported the temperature of 595 K.[13] As described in §3.2.2 and 3.2.3, we observed flat terraces at a growth temperature of 550 K. Formation of these atomically flat films is consistent with the relation of surface diffusion and growth temperature to the mechanism of epitaxial growth. That is, an atomically flat terrace can be formed when the growth temperature is sufficiently high, i.e., above a critical temperature where the Schwoebel barrier is overcome.[22] Thurmer *et al.* reported that this critical temperature is about 400 K.[9] Above this temperature, the atoms can diffuse not only in an upward but also in a downward direction on the step edges forming flat terraces.

The growth morphology of the Fe thin film on the cleaved substrate, shown in Fig. 4(d), is not in agreement with previous studies, in which the growth temperature of 383 K is too low to form atomically flat terraces films. This most likely due to the surface diffusion related with growth temperature, but is also influenced by the surface structure of the cleaved surface.

Concerning the presence of regions which exhibit very narrow terraces separated by monolayer height steps on the cleaved substrate, this kind of surface structure can be expected to behave as a vicinal-like surface that can provide high mobility to the Fe atoms to diffuse along step edges. Thus, with a growth temperature of 383 K, that is 120 degrees lower than the growth temperature of a previous study,[9,13] Fe atoms grown on this vicinal-like surface should have a high enough mobility to diffuse along step edges, forming atomically flat films as good as those formed at higher temperatures. The growth mode of Fe film in this region can be considered as layer by layer growth. Since our film thickness is 25 ML, we expect that this film grows in layer by layer mode not only in the first monolayer thickness, but at least up to 25 ML.

3.3 Magnetic properties

Figure 5 shows typical hysteresis loops of Fe thin film grown on the annealed substrate at 383 K which was measured by SQUID. A magnetic field was applied in the plane direction of the film surface. Figures 5(a) and 5(b) shows hysteresis loops along an easy axis (Fe[100]) and a hard axis (Fe[110]), respectively. All films grown on both annealed and polished substrates with growth temperature below or at 550 K show a similar hysteresis, while only the films grown on the cleaved substrate below or at 383 K show a similar behavior.

Jordan *et al.* reported a low coercivity of less than 10 Oe for 5 nm thick Fe on MgO(001).[13] They also found secondary jumps in a hard axis hysteresis loop at = 400 Oe, which is the result of biaxial magnetic anisotropy. Previously, similar loops have been found by Daboo *et al.* in Fe/GaAs[23] and Postava *et al.* in Fe/MgO.[21] We found a low coercivity of about 8 Oe and secondary jumps of about 300 Oe.

Figure 6 shows hysteresis loops of Fe thin films grown on annealed substrates at a growth temperature of 593 K (line) and 633 K (dotted line). A magnetic field was applied along the easy axis. Although the film grown at 593 K is still continuous, the hysteresis loop shows a degradation of magnetic properties. The film saturates at about 300 Oe of applied magnetic field, which is higher than in the case of other continuous films. The loop also shows less magnetic anisotropy. It is interesting that increase in the growth temperature from 550 K to 593 K, which broke the flat terrace structures, also degrades the magnetic properties. This finding is not in agreement with the previous study that reported that all continuous films showed similar hysteresis loops with low coercivity and a biaxial anisotropy.[13] The interesting point is also that Fe film grown on a cleaved substrate at 493 K shows a similar behavior to film grown on the annealed substrate at 593 K. This should be concerned to the discontinuous film on the cleaved substrate at this growth temperature.

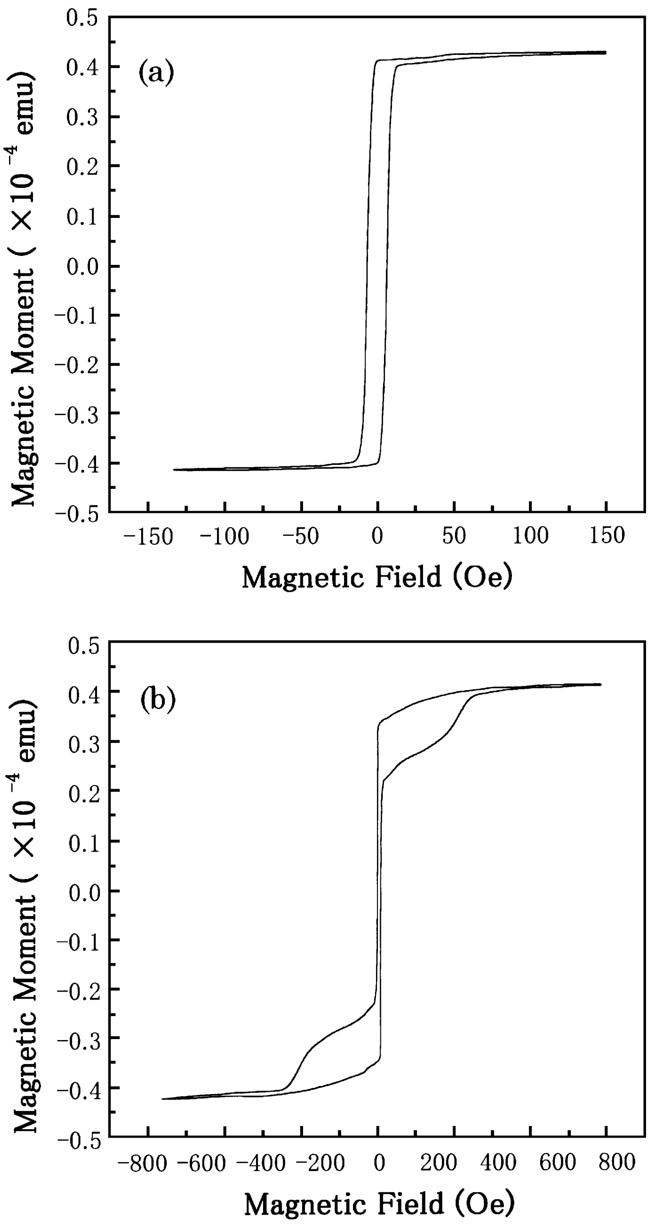

Fig. 5. Magnetic moment curves as a function of applied field of a 25 ML Fe thin film epitaxially grown on an annealed MgO(001) substrate for growth at 383 K, measured by SQUID. Measurements were carried out at 300 K. A magnetic field was applied in the plane along the (a) Fe [100] and (b) Fe[110] directions.

Superparamagnetic behavior of Fe films grown at 700 K has already been reported by Park *et al.*[18] Jordan *et al.* also reported paramagnetic behavior of Fe films grown at 695 K.[13] We found that Fe thin film grown at 633 K has superparamagnetic-like behavior as shown in Fig. 6. All the discontinuous films grown at 633 K on each substrate have a similar behavior. It will be interesting to explore the correlation between the evolution of film structure grown at or above the optimum temperature available to produce the most flat films and the magnetic properties, however, this is beyond the scope of this study.

4. Conclusions

We have investigated the relation between growth morphology and magnetic properties of bcc-Fe(001) films, which were epitaxially grown on MgO(001) substrates with various

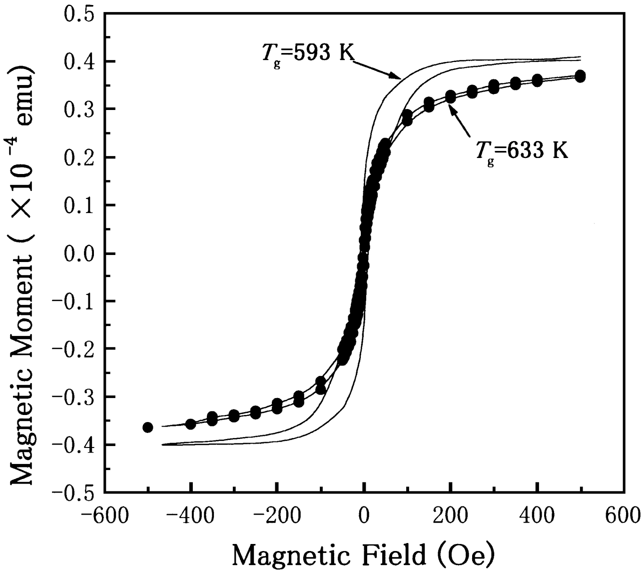

Fig. 6. Magnetic moment curves as a function of applied field of a 25 ML Fe thin film epitaxially grown on annealed MgO(001) substrates for growth at 593 K (lines) and 633 K (dotted lines), measured by SQUID. Measurements were carried out at 300 K. A magnetic field was applied in the plane along the easy axis Fe[100] direction.

surface structures at various growth temperatures. We found that the presence of steps on the substrates played an important role in the growth morphology and the magnetic properties of the Fe thin films. It also revealed that annealing for a short period of time could provide a high quality substrate for producing high quality of Fe thin films.

Compared to the polished substrates, Fe thin films grown on the annealed substrate at a growth temperature of 550 K formed atomically flat films. The monolayer height steps on the annealed substrate played an important role in improving the mobility of the atoms to diffuse along step edges to form atomically flat films. However, the presence of monolayer height steps on the annealed substrate did not significantly change the magnetic properties.

The nonuniform step-terrace structures on the cleaved substrates induced the nonuniform growth morphology of the Fe thin films. It was very difficult to obtain a flat Fe surface in the thickness of 25 ML, because the films became discontinuous at a growth temperature at or above 493 K, which is lower than that of the films grown on the other substrates. This discontinuity is caused by the presence of high steps on the cleaved substrate that break the continuity of the Fe thin films since the atoms can diffuse not only in the upward but also in the downward direction along step edges. This discontinuous film was found to show less magnetic anisotropy.

The Fe thin films grown on a polished substrate became discontinuous at a growth temperature above 550 K, while films on an annealed substrate became discontinuous at a temperature above 593 K. It is interesting that the continuous film grown on the annealed substrate at a temperature of 593 K has a different magnetic behavior from that exhibited by the other continuous films. This film has less magnetic anisotropy, however, the other continuous films have low coercivity and biaxial magnetic anisotropy, which is consistent with the previous work.[13] This characteristic of less magnetic anisotropy was similar with that of the discontinuous film grown on the cleaved substrate at a growth temperature of 493 K. It is expected that the degradation of magnetic properties on this continuous film is caused by the degradation of the film flatness when the growth temperature is increased from 550 K to 593 K, however, we still do not have an enough information to confirm this.

Considering that the spin-polarization of surface electron states strongly depends on the local arrangement of atoms, straightness of steps and the terrace width should play an important role in determining this magnetic property. Thus, the flat Fe(001) thin film with defined terraces and steps structures, such as the film grown on the annealed substrate at a growth temperature of 550 K, is expected to be suitable as a test sample for spin-polarized tunneling experiments.

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