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RHEED study of the texture in polycrystalline films of magnetite grown on oxidized silicon surface

Vyacheslav V. Balashev[1,2,a*], Victor A. Vikulov[1,b], Tatiana A. Pisarenko[1,2,c], Vladimir V. Korobtsov [1,2,d]

[1]Institute of Automation and Control Processes, FEB RAS, 5 Radio St., Vladivostok, 690041, Russian Federation

[2] Far Eastern Federal University, School of Natural Sciences, Office L457, FEFU Laboratory, 6 Ajax St., Russian Island, Vladivostok, 690922, Russian Federation

[a]balashev@mail.dvo.ru, [b]vikulov@iacp.dvo.ru, [c]tata_dvo@iacp.dvo.ru, [d]korobtsov@iacp.dvo.ru

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Abstract. Polycrystalline films of magnetite (Fe_3O_4) with thickness of 75 nm were grown on SiO_2/Si(001) surface by reactive deposition of Fe in O_2 atmosphere. The growth of Fe_3O_4 films was carried out with the different oxygen pressures. The structure of films was monitored by reflection high-energy electron diffraction (RHEED) during the film growth. It was found that there is a range of oxygen pressure in which the growth of only textured Fe_3O_4 film takes place. Reactive deposition of Fe at lower oxygen pressure results in a growth of Fe_3O_4 film without a texture. In contrast at the deposition with higher oxygen pressure the texture in the Fe_3O_4 film remains but the appearance of hematite (α-Fe_2O_3) is observed.

Introduction

Ferrimagnetic Fe_3O_4 is a well known half-metal with a high Curie temperature of ~850 K. It has a great application potential in spin electronic devices due to expected 100% spin polarization according to band structure calculation [1]. Fe_3O_4 may be used as an injector of spin-polarized conduction electrons in a semiconductor for such hybrid structures as ferromagnetic/semiconductor. As consequence a lot of studies were devoted to growth of Fe_3O_4 films on silicon substrates [2, 3]. However, it was found the formation of iron silicide and amorphous oxide on bare Si surface at an initial stage of Fe_3O_4 film growth [4]. Therefore different buffer layers [5, 6] were applied at the Fe_3O_4/Si interface to avoid the formation of detrimental layer. The growth of Fe_3O_4 film on SiO_2 buffer one was investigated as well [7, 8, 9]

Analysis of various studies has shown that the growth of polycrystalline magnetite films on SiO_2 layer with the grain preferred orientation (texture) or without one can take place. It is worth to note that the texture in Fe_3O_4 films depends on the deposition technique and growth conditions. As an example the pulse laser deposition (PLD) from α-Fe_2O_3 target leads to the film growth both with strong (111)-texture [2] and (311)-one [8]. There was no texture in Fe_3O_4 films deposited by reactive rf magnetron sputtering using an Fe target and Ar-O_2 mixture gas [10, 11]. On the other hand in the work of Zhang et al. [12] the deposition by the same sputtering technique resulted in (110)-, (100)-, or (111)-textures in Fe_3O_4 films depending on an oxygen partial pressure.

In our previous works [13, 14] we demonstrated the possibility of Fe_3O_4 film growth with (311)-texture on SiO_2/Si(001) using the reactive deposition technique. The texture was identical in the temperature range of preparation (200-400^{o}C). In our experiments the deposition rate of Fe and oxygen pressure were kept constant 0.8 nm/min and 1.3×10^{-6} Torr, respectively. In this study we investigated the influence of oxygen pressure on structural properties of the deposited films.

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Experiment

The experiments were performed in an ultra-high vacuum MBE-system "Katun" equipped with 20 keV RHEED and spectral ellipsometry instruments. The base pressure of the MBE system was 1×10^{-10} Torr. The n-type Si(001) wafers of 7.5 Ω×cm resistivity were used as substrates. Before loading into the vacuum chamber these substrates were covered by an ultrathin SiO_2 layer (~1.5 nm) by immersion of them into the boiling nitric acid for 5 minutes. After loading in vacuum chamber the substrate was degassed at 500°C for 1 hour.

Fe_3O_4 films with thickness of 75 nm were grown SiO_2/Si(001) surface by reactive deposition of Fe in O_2 atmosphere at substrate temperature of 300°C. Iron was thermally evaporated from an alumina crucible with a rate of 2.5 nm/min. The O_2 partial pressure for different samples was varied in the range of 1×10^{-6}-5×10^{-5}Torr. RHEED patterns were obtained in the deposition process at a glancing angle of ~0.5-1°.

Results and discussions

In Figure 1 an evolution of RHEED patterns at the film deposition is shown. During an initial stage of Fe deposition at an oxygen pressure of ~1×10^{-6} Torr the RHEED pattern has shown Debye rings typical for polycrystalline FeO film (Fig. 1*a*).

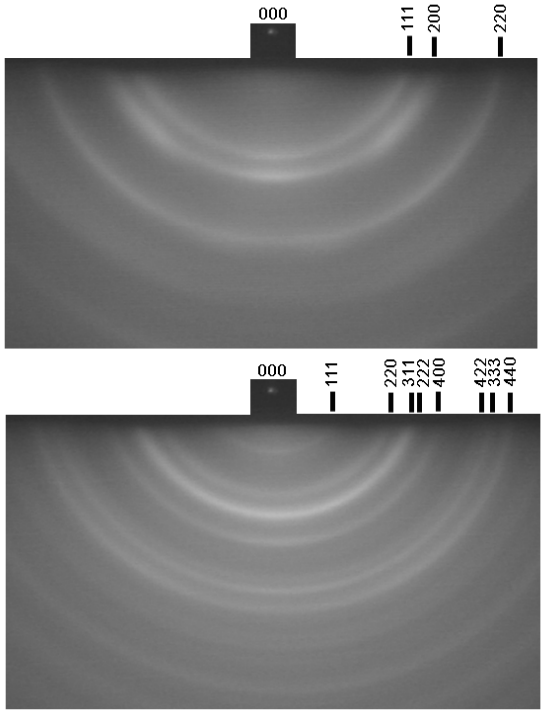


Fig. 1. *Evolution of RHEED patterns obtained in an oxygen rich environment at pressure of 1×10^{-6}Torr after the Fe deposition of: a) d_{Fe}=18 nm; b) d_{Fe}= 37 nm*

Positions of most intensive rings (marked by vertical lines in Fig. 1*a*) are in accordance with the inverse values of the interplanar distances ($1/d_{hkl}$) for reflections (111), (200) and (220) of the FeO cubic lattice with rock salt structure. The deposition of more than half of iron thickness (≥19 nm) leads to disappearance of FeO rings and the appearance of a new group of rings (Fig. 1*b*), which conserved invariable to the Fe deposition completion. Positions of the new Debye rings agree the positions of theoretical ones (marked by vertical lines in Fig. 1*b*) calculated for the inverse spinel crystal structure of magnetite [15]. The uniform intensity of rings evidences about random orientation of Fe_3O_4 crystallites in the deposited film, whereas their high sharpness is interpreted by a large size of crystallites. Because FeO rings were observed at initial stage of the film deposition

the presence of Fe grains in addition to Fe_3O_4 ones can not be excluded [16]. In our case its presence can be confirmed by a high remanence M_r/M_s=0.85 and by a saturation magnetization up to 800 emu/cm^3, which considerably exceeds a saturation magnetization for the magnetite (Fig. 2, *curve 1*).

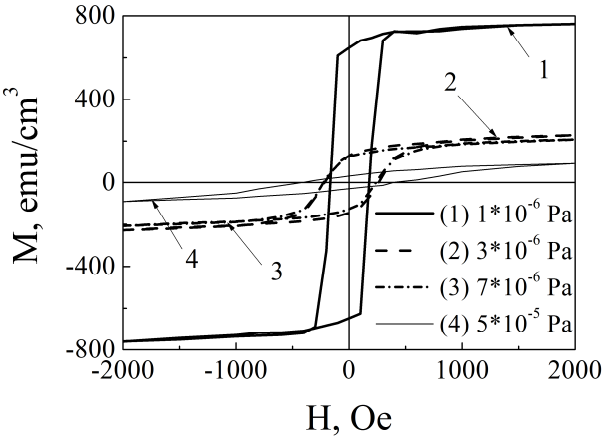


Fig. 2 *Magnetic hysteresis loops of films deposited at an oxygen pressure of: 1 - 1·10^{-6} Torr, 2 - 3·10^{-6} Torr, 3 - 7·10^{-6} Torr, 4 - 5·10^{-5} Torr.*

The deposition at higher oxygen pressure, 3×10^{-6} Torr (Fig. 3*a*) and 7×10^{-6} Torr (Fig. 3*b*), resulted in a change of the formation mechanism of Fe_3O_4 film. In this case RHEED pattern shows arcs, which are situated along the positions of Debye rings corresponding to polycrystalline film of the magnetite.

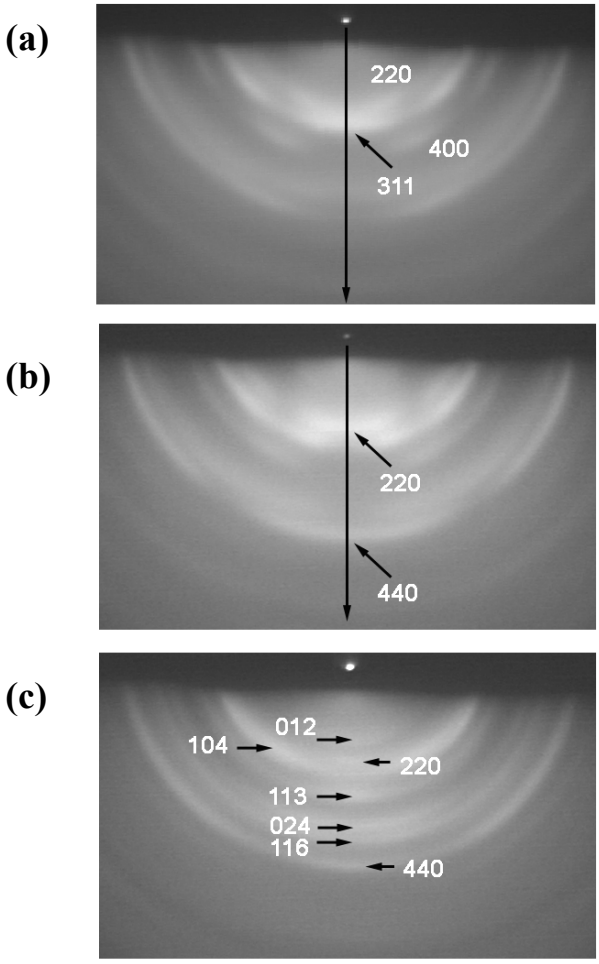


Fig. 3 *RHEED patterns obtained after the deposition of 37 nm Fe at oxygen pressure of: a) 3×10^{-6}Torr; b) 7×10^{-6}Torr and c) 5×10^{-5}Torr.*

These arcs evidence about a growth of crystallites having the preferred orientation [17]. The experimental RHEED pattern for the film obtained at pressure of ~ 3×10^{-6} Torr (Fig. 3*a*) is in a good accordance with theoretical diffraction pattern calculated for the magnetite film with (311) fiber texture [13]. The texture axis coincide with surface normal **n**, and (311) arc is located symmetrically with respect to the normal as shown by the arrow in Figure 3a. The increase of oxygen pressure up to 7×10^{-6} Torr results in formation of Fe_3O_4 film with another texture (Fig. 3*b*). Analysis of RHEED pattern shows that positions of arcs are in accordance with theoretical diffraction pattern for (110) fiber texture [18]. The (110) texture axis is also parallel to the surface normal, whereas the (220) and (440) diffraction arcs (marked by arrows in Fig. 3*b*) locate symmetrically with respect to **n**. Magnetic hysteresis loops for films obtained at oxygen pressure of $3\cdot10^{-6}$ and$7\cdot10^{-6}$ Torr (Fig. 2, *curves 2 and 3*) are typical for single domain Fe_3O_4 film, that confirm uniform phase composition.

On further oxygen pressure increase up to ~$5\cdot10^{-5}$ Torr as can be seen in Figure 3*c* the new group of Debye rings in addition to ones relating to magnetite film with (110) texture was observed. It was found that radii of these additional rings are in accordance with $1/d_{hkl}$ for crystal lattice of hematite (α-Fe_2O_3). Most intensive rings are observed for (012), (104), (024), (113) and (116) Bragg reflections marked in Fig. 3*c* by arrows directed to the right. Similar effect was observed for films obtained by Fe reactive deposition at higher oxygen pressure of $7.5\cdot10^{-4}$ Torr [19]. As it was shown in Ref.[19] the reactive Fe deposition at oxygen pressure of $7.5\cdot10^{-4}$ Torr results in single-phase hematite film without magnetite fractions.

Magnetic characteristics of this film (Fig. 2, *curve 4*) also evidence about two-component composition of iron oxide film. The presence of α-Fe_2O_3 grains in the film results in considerable decrease of the saturation magnetization and in increase of coercitivity.

Figure 4 shows AFM images of the surface structure of the magnetite films with (311) and (110) texture. From the analysis of these images it has been established that average size of crystallites in films with (311) (Fig. 4*a*) and (110) texture (Fig. 4*b*) is ~30 and 40 nm, respectively. Hence, at oxygen pressure increase from 3×10^{-6} to 7×10^{-6} Torr, in addition to a texture change from (311) to (110), the increase of crystallites size is observed. The effect of oxygen pressure on the texture and the crystallite size was also marked in Ref. [12] at magnetron sputtering of Fe.

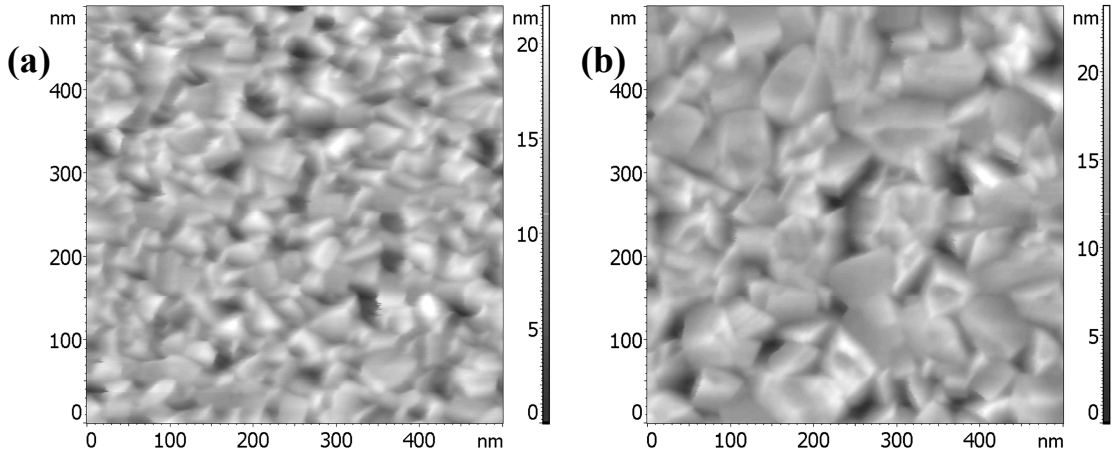


Fig. 4 *AFM image of the surface structure of the magnetite films obtained at oxygen pressure of: a) 3×10^{-6}Torr and b) 7×10^{-6}Torr.*

The change of the film texture depending on the gas flux was observed at the growth of δ-TaN films [20]. Authors of this work have shown that the texture change with the increase of reactive gas flux is concerned with intensive processes of dissociation and chemisorption on low-diffusivity planes (with low potential energy). As a result atoms of the gas become nucleation centers and interact with migrating Ta atoms, whereas the chemisorption on high-diffusivity planes (with high potential energy) remains at constant value. Thus, the crystallite planes with low energy have higher

growth rate. In consequence of this for the fcc (face-centered cubic) structure there should be the following sequence of texture transition: (111) → (100) → (110) → (311).

However, in the case of the magnetite film growth, at the increase of oxygen pressure it was observed transition from (311) texture to (110) one in our work and from (110) to (111) in Ref.[12], that is evidence of the inverse sequence of the texture change. It is possible that at the reactive Fe deposition in O_2 atmosphere there is another growth mechanism in comparison with one proposed in Ref.[20]. In our case it is supposed that adsorbed O_2 molecules react with Fe atoms to create the metastable molecular species with some surface diffusion mobility. The change of the interaction process of Fe with O_2 towards the formation of oxidized iron metastable species on the surface should result in a change of the Fe_3O_4 film growth mechanism. The corroboration of this suggestion can be seen in Ref.[21], where it was found that at the use of pulse laser deposition (PLD) the formation of the crystalline Fe_3O_4 films is determined by the surface migration process of $Fe_{1-x}O$ species laser-evaporated from both Fe_3O_4 and α-Fe_2O_3 targets. We suppose that at the oxygen pressure increase the concentration of metastable iron oxide species rises, while growth rate of crystallites in a direction normal to planes with more high potential energy, like (111) plane [12] and (110) one (this work), becomes higher. We suggest that the Fe_3O_4 film growth with only (111) texture using PLD [2, 22], regardless of the substrate type, is caused by a high content of metastable iron oxide species in ablation plasma.

Conclusions

Using RHEED technique and magnetic hysteresis loops we investigated the structure and the composition of thin magnetite film grown on SiO_2/Si(001) by reactive deposition of Fe in O_2 atmosphere. It was established that the magnitude of oxygen pressure affects the growth mechanism of polycrystalline Fe_3O_4 films and the orientation of crystallites in them. In the case of low O_2 pressure at the initial stage of deposition it was observed the formation of FeO crystallites with the following transition to Fe_3O_4 growth. Grown at these conditions the polycrystalline film of magnetite has no texture, but one contains some amount of iron. At higher values of the oxygen pressure, Fe_3O_4 crystallites are nucleated at the beginning of the deposition, and Fe_3O_4 film becomes textured. The magnetite films had (311) and (110) fiber textures, which axes coincide with the surface normal. It was found that the (311) texture changes to (110) one with the oxygen pressure increase. The growth of the Fe_3O_4 film at even more high oxygen pressure resulted in formation in it the crystallites of hematite.

Acknowledgements

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