n-Si(111) 기판 위에 전기증착에 의한 Fe 박막의 성장과 구조적 특성

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Growth and Structural Properties of Fe Thin Films Electrodeposited on n-Si(111)

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요 약

펄스 전기증착법에 의해 단결정 Fe 박막을 n-Si(111) 기판 위에 직접 성장시켰다. CV 분석을 통해 Fe^{2+}/n-Si(111) 계면은 쇼트키 장벽 형성에 따른 다이오드 특성을 가진다는 사실을 알 수 있었다. 또한 인가 전압에 따른 전기용량의 변화를 보여주는 Mott-Schottky chottky(MS) 관계식을 이용하여 전해질 내에서 n-Si(111) 기판의 flat-band potential(EFB)을 조사하였으며, 0.1 M $FeCl_2$ 전해질 내에서 EFB와 산화-환원 전위는 각각 -0.526 V 과 -0.316 V 임을 알 수 있었다. Fe/n-Si(111) 계면반응 시, Fe 증착 초기 단계에서의 핵 형성과 성장 운동학은 과도전류 특성을 이용하여 조사하였으며, 과도전류 특성을 통해 Fe 박막의 성장모드는 "instantaneous nucleation and 3-dimensional diffusion limited growth"임을 얼 수 있었다. 주파수가 300 Hz, 최대 전압이 1.4 V인 펄스 전압을 이용하여 n-Si(111) 기판 위에 Fe를 직접 전기증착 시켰으며, 형성된 Fe 박막은 단결정 α-Fe로 Si 기판 위에 α-Fe(110)/Si(111)의 격자 정합성을 가지고 성장하였음을 XRD 분석을 통해 확인하였다.

ABSTRACT

Single crystal Fe thin films were grown directly onto n-Si(111) substrates by pulsed electrodeposition. Cyclic Voltammogram(CV) indicated that the Fe^{2+}/n-Si(111) interface shows a good diode behavior by forming a Schottky barrier. From Mott-Schottky (MS) relation, it is found that the flat-band potential of n-Si(111) substrate and equilibrium redox potential of Fe^{2+} ions are -0.526 V and -0.316 V, respectively. The nucleation and growth kinetics at the initial reaction stages of Fe/n-Si(111) substraste was studied by current transients. Current transients measurements have indicated that the deposition process starts via instantaneous nucleation and 3D diffusion limited growth. After the more deposition, the deposition flux of Fe ions was saturated with increase of deposition time. From the as-deposited sample obtained using the potential pulse of 1.4 V and 300 Hz, it is found that Fe nuclei grows to three dimensional(3D) islands with the average size of about 100 nm in early deposition stages. As the deposition time increases, the sizes of Fe nuclei increases progressively and by a coalescence of the nuclei, a continuous Fe films grow on the Si surface. In this case, the Fe films show a highly oriented columnar structure and x-ray diffraction patterns reveal that the phase α-Fe grows on the n-Si(111) substrates.

키워드

α-Fe, growth mode, Si(111), electrodeposition, metal/semiconductor interface

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Ⅰ. INTRODUCTION

Metallization of semiconductor surfaces is an important technological process for the formation of Schottky barriers, ohmic contacts, and diffusion barriers in the fabrication of integrated circuits. In addition, the fabrication of nanocrystalline materials on Si substrates is also important subject because of their fundamental interest and their potential applications in the field of quantum devices. In particular, the growth of thin ferromagnetic films (Fe, Ni, Co) on the semiconductor substrates is of great important because of its future applications in si-based magnetic devices.

The growth of Fe thin films as well as Fe-silicides on Si substrates has also been extensively carried out due to their potential applications in the electromagnetic or optoelectronic devices, and Si-based integrated circuits[1,2,3,4]. In general, the phase formation kinetics and the stoichiometry of Fe:Si and crystallography largely depend on the deposition method and coverage. It is well known that the Fe/Si interface is very reactive and unstable at the initial stages of Fe deposition in vacuum. Even at room temperature (RT), a complex interfacial reaction causes various phases such as Fe clusters, FeSi, Fe_3Si, and $FeSi_2$ to be formed on the Si surface.[5,6,7]

Vacuum-based techniques have been generally used to deposit metal thin films onto Si substrates. Recently, electrodeposition has received renewed attention as it is an efficient, inexpensive, and convenient method to grow metal films on Si substrates.[8,9,10,11,12,13] In contrast to the vacuum-based deposition, electrodeposition is a nonequilibrium deposition technique which has various parameters such as the types of deposition potential, the concentration of metal ions, and the pH of aqueous electrolyte solution, etc. Therefore, we can expect that Fe thin films grown by an electrodeposition show very different structural characteristics compared with that obtained by vacuum-based techniques.

In this paper, we report on the growth of highly oriented columnar structured Fe thin films on n-Si(111) substrate without any buffer layer by a pulsed electrodeposition technique. The potential-step experiments were carried out to investigate the growth kinetics of Fe nuclei in the early deposition stages. The variations of surface morphology as functions of the deposition potential and its frequency were also investigated by field emission scanning electron microscope (FESEM). The crystallographic structure of the as-deposited Fe thin films was analyzed by X-ray diffraction (XRD).

Ⅱ. EXPERIMENTAL WORK

Phosphorous-doped n-Si(111) wafers with resistivity of 5-10 Ωcm were used as substrates. Si wafers (1 cm × 1 cm) were cleaned successively in acetone and ethanol, and then rinsed in flowing deionized water. The Si substrates were etched for 2 min in 47 % HF and then for 5 min in 40 % NH_4F solutions to remove native oxide. Details of the Si wafer cleaning procedures are found elsewhere.[14] The Si wafer was immediately transferred to the reaction cell and was used as the working electrode (cathode).

Iron rod (99.999 %) of a 3-mm diameter was used as an auxiliary electrode (anode). The electrolyte solution was prepared by dissolving $FeCl_2 \cdot H_2O$ in methanol(99.89 %). It is known that the interfacial reaction at the metal ions/ semiconductor is more stable at higher ion concentrations ($>10^{-2}$ M)[15], the concentration of the electrolyte used was 0.1 M $FeCl_2$. Generally, electrodeposition in an aqueous solution has the problems of pH control of the electrolytes as well as the hydrogen bubbles generating on the working electrodes. In order to minimize these problems, we have used non-aqueous methanol electrolyte solution. The electrical and electrochemical characteristics of the Fe^{2+}/n-Si(111) interface in the electrolyte solution were studied by CV and MS relation (C_{sc}^{-2} versus E). To investigate the nucleation and the growth behaviors by the interfacial reaction of Fe^{2+}/Si during Fe deposition, potential-step experiments were performed in the ranges from 1.0 V to 1.4 V and the resulting current transients were analyzed with the model of Scharifker and Hills[16,17] for 3D islands growth under diffusion-limited control.

All electrochemical measurements were performed with a potentiostat, BAS 100B/W electrochemical workstation. All the depositions were made in an unstirred solution at RT.

During the deposition, the reaction cell was continuously purged with high purity dry N_2 to remove oxygen in it and minimize the oxygen effects on the Si surface exposed to the electrolyte solution. In the present electrodeposition technique, the periodic potential pulses of a definite form were applied to deposit Fe on Si substrate. These potential pulses were generated from Hewlett-Packard 8116A Pulse/Function Generator. The current evolutions during the deposition were monitored using the LabView program coupled to a microcomputer. A wide range of pulse parameters was investigated to determine those favorable to the deposition of high quality films.

This paper reports only the results obtained with deposition parameters fixed at the conditions that can led to homogeneous, adherent and good crystalline Fe thin films. The current efficiency and the growth rate were estimated from the current passed and the deposition time. During the deposition, the applied potential remained constant except for a negligible ohmic potential drop at the early stage of Fe deposition. And all potentials are referred to the standard Ag/AgCl(3 M NaCl) reference electrode. The crystalline structure of the as-deposited Fe thin film was analyzed by x-ray diffraction (XRD) using Ni-filtered Cu-Kα radiation source in Θ-2Θ geometry and the surface morphology was characterized by field emission scanning electron microscope (FESEM).

Ⅲ. RESULTS AND DISCUSSION

Fig. 1 shows CV curves for the first cycle of Fe electrodeposition. The dotted curve was obtained by using n-Si(111) wafer as a working electrode and the solid one was obtained by using bulk Fe plate as a working electrode. The deposition rate of metal on semiconductor substrate in an electrodeposition is very sensitive to the relative position of the equilibrium redox potential ($E_{Fe/Fe2+}$), which corresponds to the potential at zero current density in the solid curve of Fig. 1. The value of $E_{Fe/Fe2+}$ is measured to be -0.316 V from Fig. 1. This has also been confirmed by measuring the open circuit potential of the Fe plate electrode in the same electrolyte solution. The dotted curve shows a good diode behavior,

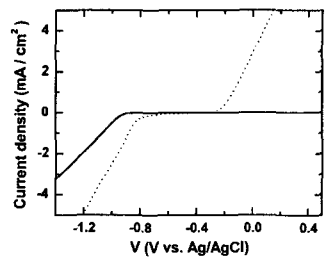

Fig. 1. CV curves with respect to the n-Si(111) substrate (solid line) and bulk iron rod(dotted line) used as a working electrode. The scan rate was 20 mV/s.
그림 1. (111)면을 가진 n형 실리콘 기판(실선)과 벌크 Fe(점선)를 작업전극으로 사용할 경우에 대한 순환전류전압 곡선. 전압증가율은20 mV/s 이었다.

which implies that the Fe/n-Si(111) interface forms a Schottky contact. Thus, the reduction process of Fe^{2+} ions is largely irreversible. This indicates that the deposition process of Fe^{2+} ions is more dominant than the dissolution process from the surface of n-Si(111) substrate.

Since the reciprocal of the square of the capacitance of the semiconductor is linearly related to the electrode potential and independent of the measuring frequency,[18,19] we measured the capacitance of the Fe^{2+}/n-Si(111) contact. The C_{sc}^{-2} versus E plot (MS plot) for n-Si(111) electrode in contact with the electrolyte solution measured at a frequency of 5 kHz is shown in Fig. 2. The donor density, N_d, of n-Si(111) substrate which is obtained from the slope of the best fit line (dotted line) is about 5.3×10^{14} cm^{-3}. This value agrees well with the value obtained from Hall effect measurement.

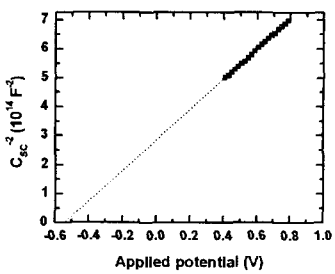

Fig. 2. MS plot for the 0.1 M $FeCl_2$ electrolyte/n-Si(111) contact measured at a frequency of 5 kHz. Rectangular symbol and the dotted line represent the measured data and a linear fit to the data, respectively.
그림 2. 5 kHz의 주파수로 측정한 0.1 M $FeCl_2$ 전해질용액과 (111)면을 가진 n형 실리콘 기판의 접촉에 대한 Mott-Schottky 그림. 사각형 기호와 점선은 측정데이터와 이 데이터에 대한 이론적 결과를 각각 나타낸다.

The flat band potential, which is related with the band bending of n-Si(111) surface conduction band at zero potential, is obtained from the intercept of the best fit line. The value of the flat band potential E_{fb}, of Si substrate for the Fe^{2+}/n-Si(111) contact is about to be -0.526 V. In this case, since the redox potential $E_{Fe/Fe2+}$ is located in the band gap and is more positive than the flat band potential E_{fb}, a depletion layer can be formed at the surface of n-Si(111) substrate. In order to overcome this potential barrier, negative potential should be applied. In a semiconductor-electrolyte solution contact, the driving force ($\triangle G^0$)[20] for the interfacial reactions is given by

$$\Delta G^0 = q E_{fb}^0 - kT \ln(N_C / N_d) - q E_{Fe/Fe^{2+}} = q E_{fb} - q E_{Fe/Fe^{2+}} \quad (1)$$

where, N_C and N_d are the effective density of states in the conduction band and the donor density of the Si substrate, respectively. k and T are the Boltzmann constant a nd the temperature of the solution. In the case of Fe deposition the driving force, $\triangle G^0$ /q, for the interfacial electron transfer between Fe^{2+} ion and n-Si(111) substrate is -0.21 V.

Fig. 3 shows a series of curves representing the variation of the current densities as function of times obtained by the potential step experiment in the ranges from 1.0 V to 1.4 V. In all curves (except the curve for 1.0 V), it is shown that the current density increases rapidly as Fe begins to nucleate on the Si substrate, and then shows an asymptotically decaying

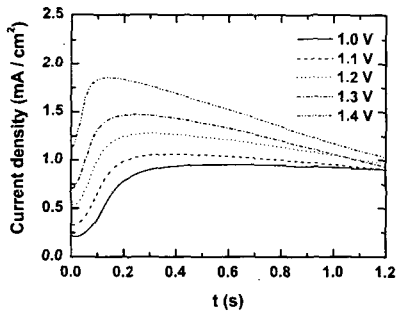

Fig. 3. Current transients for Fe deposition on n-Si(111) substrates in the potential ranging from 1.0 to 1.4 V.

그림 3. 1.0 V에서 1.4 V 사이의 전압 영역에서 (111)면을 가진 n형 실리콘 기판 위에 Fe를 증착하는 경우의 과도전류 특성 곡선.

behavior after reaching the maximum values. Accordingly, it can be concluded that the nucleation rate depends significantly upon the applied potentials and that Fe nucleation on the Si surface proceeds more rapidly when the applied potential increases.

These features are qualitatively consistent with the theoretical 3D nucleation followed by diffusion limited growth model suggested by Scharifker and Hills[16,17]. According to this model, there are two nucleation modes: the instantaneous nucleation and the progressive nucleation. In the instantaneous nucleation mode, all available reaction sites of the substrate are activated simultaneously. On the other hand, in the progressive nucleation mode, the number of nuclei formed initially on the surface is less than the possible maximum number of nucleation sites and the number of nuclei grows progressively. For these two modes, the current densities can be expressed by equations (2) and (3), respectively.

$$i(t) = \frac{zFCD^{1/2}}{\sqrt{\pi t}} \left[1 - \exp\left(-N_0 D (8\pi^3 C V_m)^{1/2} t\right)\right] \quad (2)$$

$$i(t) = \frac{zFCD^{1/2}}{\sqrt{\pi t}} \left[1 - \exp\left(-\frac{2}{3} K_n N_0 D (8\pi^3 C V_m)^{1/2} t^2\right)\right] \quad (3)$$

where, z is the valency of the metal ion, F is Faraday's constant, N_0 and D are the nucleus density and the diffusion coefficient, C is the Fe^{2+} ion concentration in the electrolyte solution, K_n is the nucleation rate and V_m is the molar volume.

The measured current transients are presented in Fig. 4, where the current has been normalized to its maximum value, i_{max}, and the time to the duration at which the current density reaches its maximum value, t_{max}. The solid line and the dotted line in Fig. 6 represent the predictions of the model for instantaneous and progressive nucleation, respectively. Although the experimental data follows neither prediction exactly, it can be seen that in the initial stage of Fe deposition the instantaneous nucleation model offers the better match of the

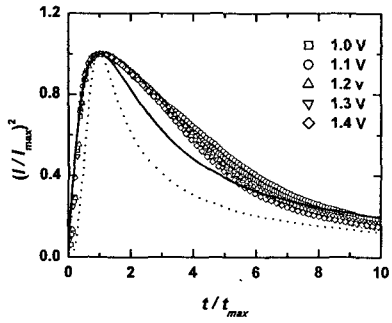

Fig. 4. Normalized $(i/imax)^2$ vs t/tmax plots obtained from the experimental data in Fig. 2. The dotted and the solid line represent the theoretical progressive and instantaneous nucleation modes, respectively.

그림 4. 그림 2의 데이터로부터 얻은 규격화된 (i/imax)2 와 t/tmax 사이의 그림. 점선과 실선은 이론적으로 제안된 progressive nucleation mode(점진적 핵형성 모드)와 instantaneousnucleation mode(순간적 핵형성 모드)를 각각 나타낸다.

two. For instantaneous nucleation mode, N_0 and D can be expressed as follow:

$$N_0 = 0.0652 \frac{1}{(8\pi CV_m)^{1/2}} \frac{(zFC)^2}{i_{max}^2 \cdot t_{max}^2} \quad (4)$$

$$D = \frac{i_{max}^2 \cdot t_{max}}{0.1629(zFC)^2} \quad (5)$$

From above relations, N_0 and D evaluated for each potential are presented in Fig. 5. It is expected that these values are meaningful only for the initial deposition stages because the experimental curves deviate from both theoretical curves at a long deposition times. As shown in Fig. 5, it is found that N_0 and D have nearly constant values and also have an order of ~10^{10} cm^{-2} and ~10^{-9} cm^2s^{-1} in the ranges from 1.0 V to 1.4 V, respectively. Compared with the values of ~10^{-5} ~10^{-6} cm^2s^{-1} in references,[21] the small D values in this work indicate a small concentration gradient near the each nuclei and also can be considered as the more stable nuclei growth. Moreover, the higher N_0 values indicate that Fe nucleates according to instantaneous mode where all reaction sites are activated at the start of the deposition.

To investigate the growth behaviors of Fe nuclei as functions of the deposition potential and its frequency, the samples were prepared by the various parameters. Fig. 6 shows

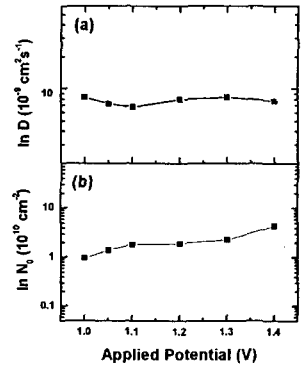

Fig. 5. N_0 and D versus applied potential. N_0 and D were estimated with the theoretical model and experimental data.

그림 5. 증착전압에 따른 N_0 와 D. N_0 와 D는 이론적 모형과 실험 데이터로부터 평가 되어졌다.

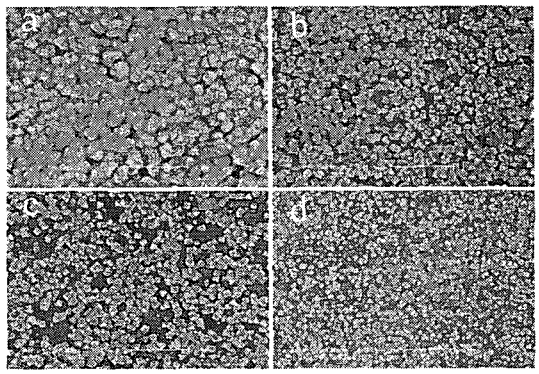

Fig. 6. FESEM images for as-deposited samples grown at the different frequencies: (a) 20 Hz, (b) 300 Hz, (c) 600 Hz, and (d) 900 Hz. (all scales are 1×1 μm^2.)

그림 6. 각기 다른 주파수에서 성장시킨 시료들에 대한 전계방출 형주사전자현미경 영상: (a) 20 Hz, (b) 300 Hz, (c) 600 Hz, and (d) 900 Hz. (각 영상의 크기는 1×1 μm^2 이다.)

the FESEM surface images of the as-deposited samples obtained at each frequency of applied potential: (a) 20 Hz, (b) 300Hz, (c) 600 Hz and (d) 900 Hz. From Fig. 6, it is found that the size of Fe nuclei decreases with increasing the frequency of the applied pulse potential and in all frequencies expect for a case of 20 Hz, the average sizes of Fe nuclei are about 120 nm(300 Hz), 86 nm(600 Hz) and 59 nm(900 Hz), respectively. In a case of 20 Hz, Fig. 6(a) shows that the nuclei size increases due to the coalescence of individual Fe nucleus and as a result, nearly continuous Fe films with higher surface roughness are formed. From Fig. 6, we can observe that as-deposited sample obtained at 300 Hz shows a good coverage and uniform distribution of Fe nuclei among them.

Fig. 7 shows the FESEM surface images of the as-deposited samples obtained at each applied potential: (a) 1.0 V, (b) 1.5 V, (c) 2.0 V and (d) 2.5 V. As shown in Fig. 7, FESEM images indicate that the size of Fe nuclei does not depend on the deposition potential and for all potential ranges, Fe nuclei has nearly the same size but different areal densities of it. With increasing the deposition potential, the surface areal density of Fe nuclei increases progressively and in a case of 2.5 V, the surface is perfectly covered with Fe nuclei but is very rough. As a result, we can expect that the deposition potential strongly affects the nuclei density.

Fig. 8 shows the XRD pattern for about 625 nm thick Fe thin film electrodeposited on n-Si(111) substrate with a maximum potential 1.4 V and the frequency of 300 Hz. Only two peaks related to the Fe film and Si(111) substrate are observed. The peak at $2\theta = 28.9^{\circ}$ is the diffraction from Si(111) plane and the peak at $2\Theta = 44.8^{\circ}$ is the diffraction from the α-Fe(110) plane. The value of the full width at half maximum of the Fe(110) diffraction peak is about 0.3°, which means that the grown film is highly oriented.[22,23] Because atomic density of the Fe(110) surface is higher than that of the Fe(100) surface by a factor of $\sqrt{2}$, the Fe(110) surface may play a role of low barriers for surface self-diffusion. Therefore, Fe(110) plane may preferentially be grown on Si(111) at low temperature.

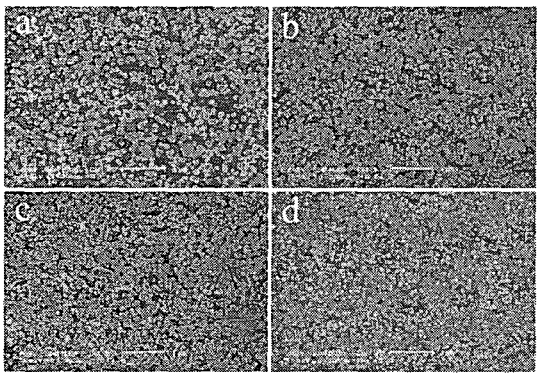

Fig. 7. FESEM images for as-deposited samples grown at the different potentials: (a) 1.0, (b) 1.5 V, (c) 2.0 V, and (d) 2.5 V. (all scales are 1×1 μm^2.)

그림 7. 각기 다른 전압에서 성장시킨 시료들에 대한 전계방출형 주사전자현미경 영상: (a) 1.0, (b) 1.5 V, (c) 2.0 V, and (d) 2.5 V. (각 영상의 크기는 1×1 μm^2 이다.)

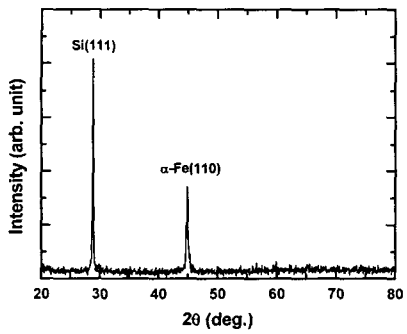

Fig. 8. XRD pattern for an as-deposited Fe film with a thickness of 625 nm grown on Si(111) substrate at RT in 0.1 M $FeCl_2$ + methanol solution.

그림 8. 실온에서 0.1 M $FeCl_2$ + 메탄올 용액을 이용하여 (111)면을 가진 실리콘 기판 위에 성장시킨 625 nm 두께의 Fe 박막에 대한 X-선 회절 모양.

Yoshimura et al.[24] investigated orientation index of Fe grains grown on a Cu plate by electrodeposition in a non-aqueous $FeCl_2$ solution. The results showed that the orientation index of the film was primarily related to the current efficiency (or current density). As the current density increases, hydrogen evolves at the cathode from a side reaction, the current efficiency decreases and the resulting crystalline structure of the film drastically changes. Films prepared at a small current density (~5 ~20 mA/cm^2) had a preferred (110) plane orientation and those prepared at a high current density (>20 mA/cm^2) were of mixed (211) and (200) plane orientations. The size of the Fe(110) grains was ~ 50 nm and that of Fe(211) and Fe(200) oriented grains was much smaller. In analogy with those results the film grown at high current efficiency with low current density has a highly preferred orientation index of Fe(110) plane with large grain size. Even if the lattice mismatch between a-Fe(110) and Si(111) planes is very large, XRD pattern shows that the a-Fe film was grown with a lattice matching relationship of α-Fe(110)//Si(111).

Ⅳ. Conclusion

Thin magnetic Fe films were electrodeposited directly onto n-Si(111) substrate. In the early interfacial reaction of Fe/n-Si(111), the deposition of Fe occurs by instantaneous nucleation of 3D islands followed by diffusion limited

growth. In this nucleation mode, all the reaction sites on the surface of Si substrate are saturated in the initial stage of Fe deposition and thus, high areal density of Fe clusters are obtained. The diffusion coefficient and the nucleus density estimated from this nucleation model have an order of ~10^{-9} cm^2s^{-1} and ~10^{10} cm^{-2}, respectively. It has been found that the size of Fe nuclei decreases with increasing the frequency of the applied pulse potential but does not depend on the deposition potential. In addition, FESEM revealed that the as-deposited samples at all potential ranges show nearly the same size but different areal densities of Fe nuclei. From the specimen prepared under the conditions of 1.4 V, 300 Hz, we have confirmed that single phase α-Fe(110) films with a high crystallinity can be electrodeposited directly on Si(111) substrate. Moreover, we suggest that Fe grows to 2D layer-by-layer plus islands growth mode (Stranski-Krastanov growth mode).

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