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Photoelectrochemical Behavior of Iron Oxide Thin Film Electrodes Prepared by Sol-Gel Method

Toshinobu YOKO, Kanichi KAMIYA*, Katsuhisa TANAKA** and Sumio SAKKA

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Iron oxide films were prepared on the silica glass substrates by sol-gel method combined with dip-coating technique. The crystallization behavior and optical properties of these films were investigated as a function of post-heating time and temperature. Photoelectrochemical and a.c. impendance measurements were made on these post-heated iron oxide film electrodes. The maximum photocurrent was obtained for the sample heated at 700°C for 2 h. An obvious break was observed for the Mott-Schottky plot of the present film electrode, indicating the presence of deep and shallow donors as in the case of the sintered body. The presence of two kinds of optical transitions with different band gap energies was also confirmed from the action spectrum. The smaller and larger ones were assigned to an indirect Fe^{3+} (3d non-bonding)—conduction band (CB) transition with E_g=2.06 eV and a direct O^{2-} (2p bonding)—CB transition with E_g=2.64 eV, respectively.

KEY WORDS: Photoelectrochemistry/ Iron Oxide/ Thin Film/ Sol-Gel Method/

1. INTRODUCTION

Utilization of solar energy is absolutely necessitated to meet future energy demands. The conversion of solar energy into electricity and/or chemical substances, for example hydrogen, using semiconductors as a photoelectrochemical electrode is also considered to be one of the most promising means. A quite number of researches have been made on this subject since Fujishima and Honda[1] succeeded in decomposing water into H_2 and O_2 using a photoilluminated TiO_2 anode without any degradation of the electrode.

The main requirements for photoelectrochemical electrodes are (1) chemical and photoelectrochemical stability and (2) high conversion efficiency[2]. TiO_2 has high chemical and photoelectrochemical stability, but low solar energy conversion efficiency only less than 4% due to its large band gap (3.0 eV). Semiconductors with small band gap, in general, lack chemical and photoelectrochemical stability. Among them, α-Fe_2O_3 with a band gap of about 2eV is an exceptional semiconductor which almost satisfies the above requirements.

Hardee and Bard[3] were the first who made photoelectrochemical studies on Fe_2O_3 films prepared by CVD form $Fe(C_5H_7O_2)_3$. Since then, several works[3-15]

横尾俊信，作花済夫：Institute for Chemical Research, Kyoto University, Uji, Kyoto-fu 611, Japan
* 神谷寛一：Department of Industrial Chemistry, Faculty of Engineering, Mie University, Tsu, Mie-ken 514, Japan
** 田中勝久：Department of Industrial Chemistry, Kyoto University, Kyoto 606, Japan

have been done using different kinds of Fe_2O_3 electrodes prepared by flux growth[4], thermal oxidation[5,9,10], r.f. sputtering[10,14,15] and sintering[6,7,11,12-14] in addition to CVD. It has been also found here that both n- and p-type Fe_2O_3 can be prepared by adding divalent and tetravalent metal oxides, respectively, as dopants[4,6,9-11]. All of them, however, show a rather low quantum efficiency and a decline in onset potential.

On the other hand, the authors have reported the preparation of semiconducting TiO_2 thin films by sol-gel method and their photoelectrochemical properties[16]. The sol-gel derives TiO_2 film electrode was found to give a large photocurrent due to the porous surface structure inherent in a gel. In the present study, sol-gel method is applied to the preparation of α-Fe_2O_3 thin films for photoelectrodes and their photoelectrochemical properties are investigated in details.

2. EXPERIMENTAL

2.1 Substrates

Synthetic silica glasses were coated with SnO_2 doped with 1 mol% Sb_2O_3 and used for substrates. SnO_2 coating films were made by a fume pyrolysis: coating solution containing 50 ml ethyl acetate, 20 ml conc. HCl, 10 g $SnCl_2$ and 0.1256 g $SbCl_3$ was sprayed onto silica glass heated at 750°C. The electrical resistance was around 50–60 $\Omega\square$.

2.2 Coating and heat treatment

Coating solutions for Fe_2O_3 films were prepared as follows. 17.7 g of iron acetylacetonate, $Fe(acac)_3$ and 60 ml of Hacac were put into 200 ml meyer flask and stirred. Then, 8.5 ml of HNO_3 (13.8N) was dropped in the solution using a pipette under stirring. The resultant solution was kept on stirring for 2–3 days prior to use.

A glass substrate obtained in section 2.1 was immersed in the solution, pulled up at a rate of 0.3 mms^{-1}, dried for 3 min and then heated at 500°C for 10 min. Fe_2O_3 films of 0.4 μm thick were obtained by repeating the above operation ten times. Post-heat treatment was carried out at temperatures between 500 and 800°C for 1–120 min in order to investigate the crystallization behavior of the as-prepared Fe_2O_3 films. The whole process is summarized in Fig. 1.

The precipitated crystalline phase was identified by an X-ray diffractometer using CoKα radiation. The crystallite size was determined from the FWHM of (104) line according to Scherrer's equation.

2.3 Absorption spectra

The absorption spectra of Fe_2O_3 films heated at various temperatures for different times were measured using a Cary Model 14 spectrometer (Varian, U.S.A.).

2.4 Photoelectrochemical measurements

Photocurrent-potential curves were recorded using a potentiostat/galvanostat model 2000 (TOHO Giken, Japan) with three electrodes system: Fe_2O_3 film working,

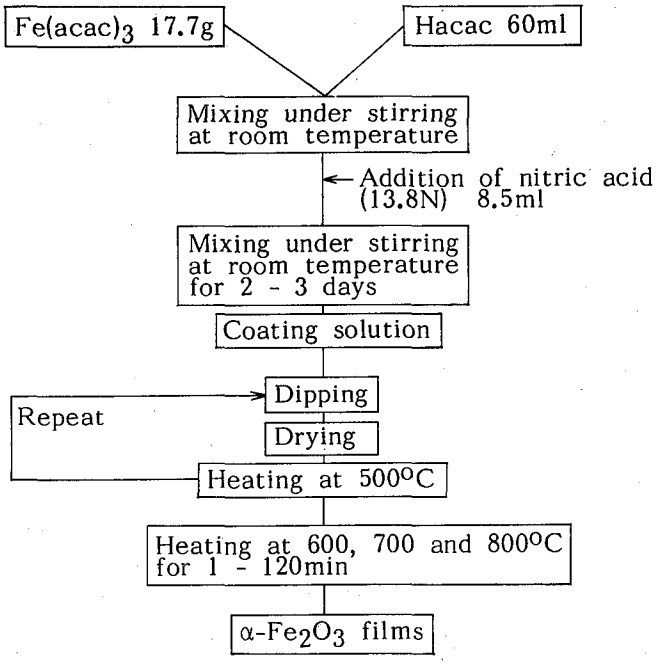

Fig. 1. Flow chart for preparing Fe_2O_3 film electrode.

platinized Pt counter and saturated calomel electrodes (SCE). Illumination was with a Xenon-arc lamp (500W). Monochromatized light in the wavenumber range from 200 to 700 nm was obtained by a monochromator model CT10 (JASCO, Japan). Impedance analysis for obtaining the Mott-Schottky plot was performed using a frequency response analyzer model S-5720B (NF, Japan) operated at 1010 Hz in conjunction with the potentiostat. Measurements and data accumulation were made with the help of a personal computer PC-9801VM (NEC, Japan) via GP-IB.

Two kinds of electrolytic solutions were used: (1) 0.2M $Na_2B_4O_7$+0.1M H_2SO_4+ 0.3M Na_2SO_4 (pH 7) and (2) 0.1M NaOH+0.3M Na_2SO_4 (pH 13).

3. RESULTS

3.1 Optical properties

The absorption spectra of Fe_2O_3 films heated at 700°C for different times are shown in Fig. 2. The spectra have two mountains and troughs in the measured wavelength region. Since these positions varied with heating time, in other words, with film thickness, they do not arise from absorption, but from the interference of a light.

Fe^{3+} and Fe^{2+} in octahedral coordination are known to show absorption bands around 800 nm and 1100 nm, respectively[17]. Both peaks, however, were not visible probably due to small absorbance compared with the interference spectrum. For all samples, irrespective of heating time and temperature, the steep rise of absorb-

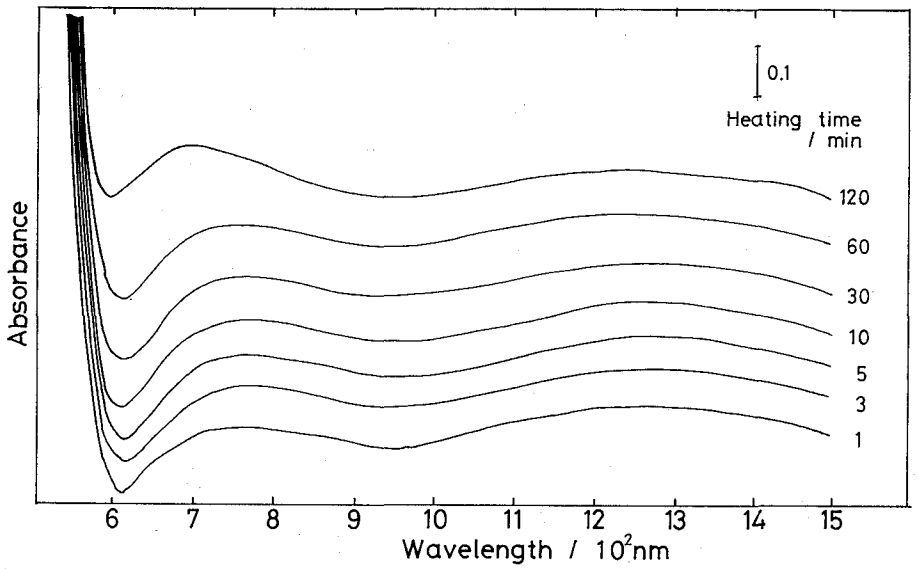

Fig. 2. Absorption spectra of Fe_2O_3 films heated at 700°C for different time.

ance was observed below 580 nm probably due to the charge transfer. The band gap energy of Fe_2O_3 films prepared in the present study was estimated from the absorption edge as about 2.1 eV.

3.2 Crystallization behavior

α-Fe_2O_3 (hematite) crystals were found to precipitate in the as-prepared Fe_2O_3 films which were heated at 500°C. Fig. 3 shows the X-ray diffraction patterns of Fe_2O_3 films forming on the SnO_2-coated silica glass which were heated at 800°C for 1 and 120 min. With increasing heating time, (110) line appeared in addition to (104) line indicating that the crystallinity was improved.

The variations of crystallite size in the α-Fe_2O_3 films heated at various temperatures with time are shown in Fig. 4. The crystallite size increased sharply at the early stage of heating up to 10–20 min and then became constant; 33 nm at 500°C and 45 nm above 600°C.

3.3 Photoelectrochemistry

Polarization curves in solutions of pH 7 and 13 for Fe_2O_3 film electrodes which were heated at 600, 700 and 800°C for various times are shown in Figs. 5a, b, 6a, b, and 7a, b, respectively. There are noticeable differences in the photocurrent and the shape of polarization curves taken in solutions of pH 7 and 13. That is, the photocurrents in neutral solution are smaller than in pH 13 solution and the polarization curves in pH 7 solution have a plateau in the potential region between 0.5 and 1V (vs. SCE). The smaller photocurrents observed in solution of pH 7 than pH 13 is clearly due to the oxygen overpotential. All the samples studied did not show a saturated photocurrent.

Figs. 8a, b and c show the time dependence of the photocurrent at several bias

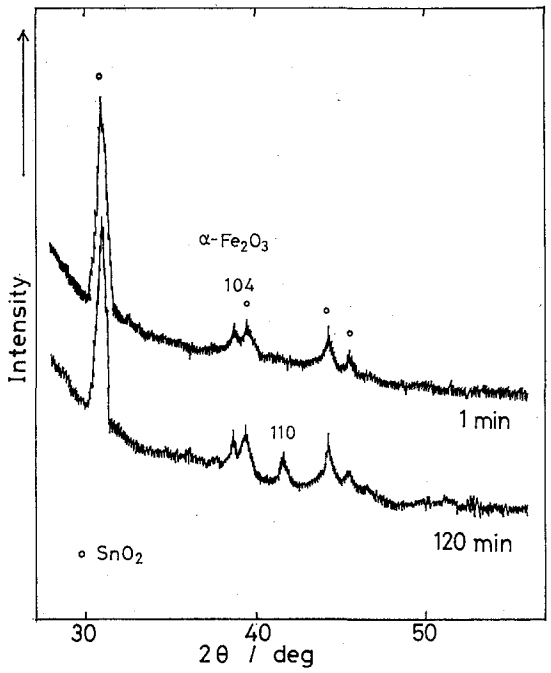

Fig. 3. X-ray diffraction patterns of Fe_2O_3 films forming on the SnO_2- coated SiO glass heated at 800°C for 1 and 120 min.

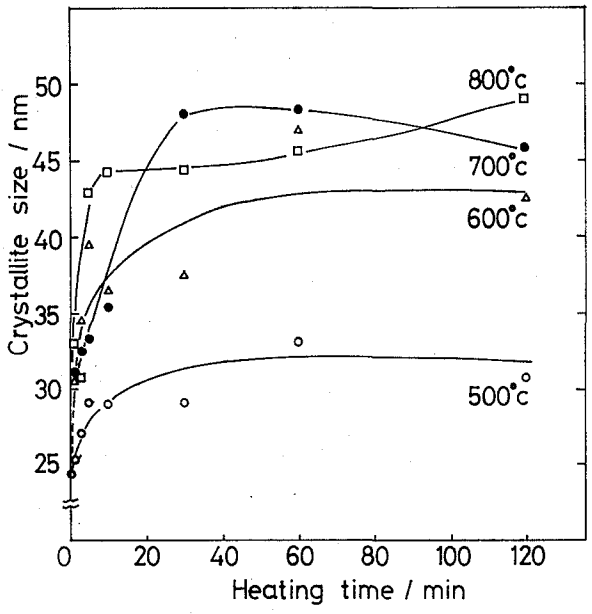

Fig. 4. Variations of crystallite size in the α-Fe_2O_3 films heated at different temperatures with time.

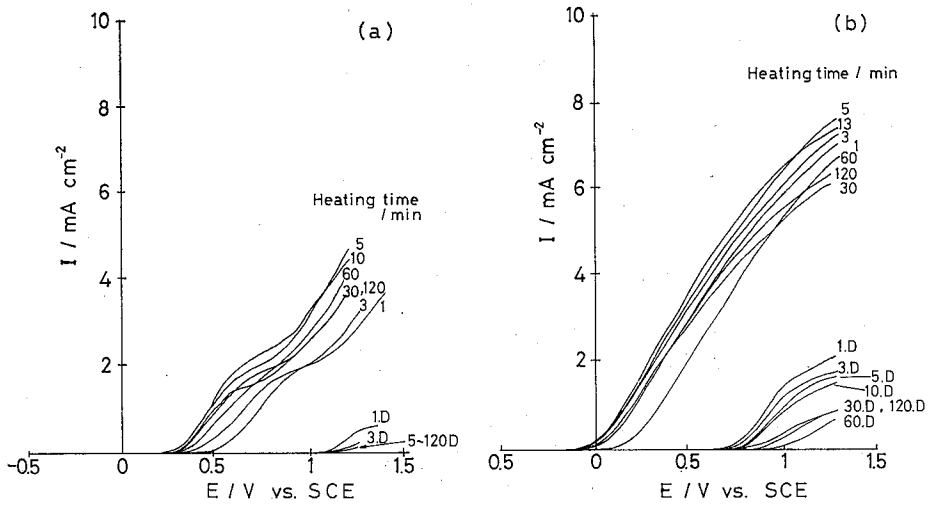

Fig. 5. Polarization curves of Fe_2O_3 film electrodes heated at 600°C; (a) pH 7 and (b) pH 13.

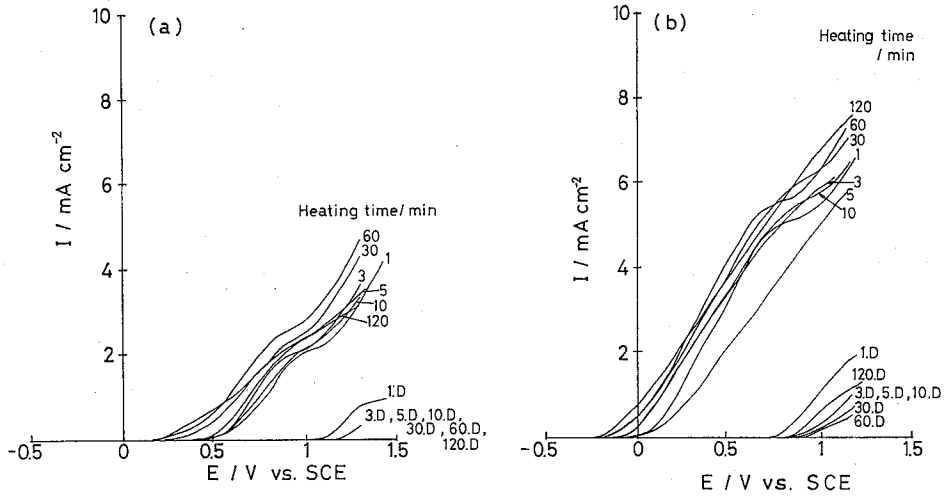

Fig. 6. Polarization curves of Fe_2O_3 film electrodes heated at 700°C; (a) pH 7 and (b) pH 13.

potentials for samples heated at 600, 700 and 800°C, respectively. At 600°C, the photocurrents first increase with heating time up to 5–10 min, decrease slightly and then attain constant values. At 700°C they gradually increase with heating time and then become constant after 60 min. On the contrary, at 800°C they decrease rapidly with heating time up to 60 min and then become almost constant.

Fig. 9 shows the action spectra taken in solutions of pH 7 and 13 for the sample heated at 600°C for 5 min. The two spectra have basically the same features, showing a maximum at 360 nm (3.45 eV) and a small maximum around 475 nm (2.61 eV) and a long tail extending up to about 600 nm (2.07 eV).

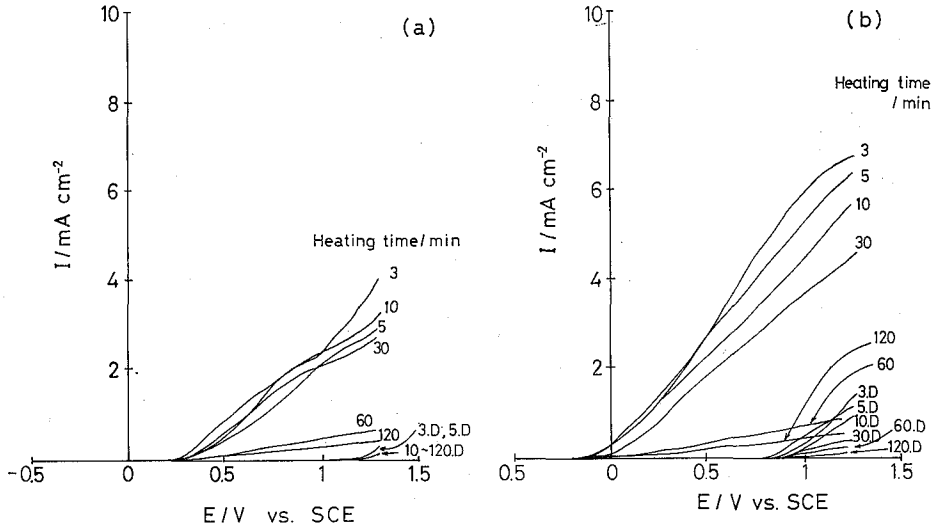

Fig. 7. Polariz Polarization curves of Fe_2O_3 film electrodes heated at 800°C; (a) pH 7 and (b) pH 13.

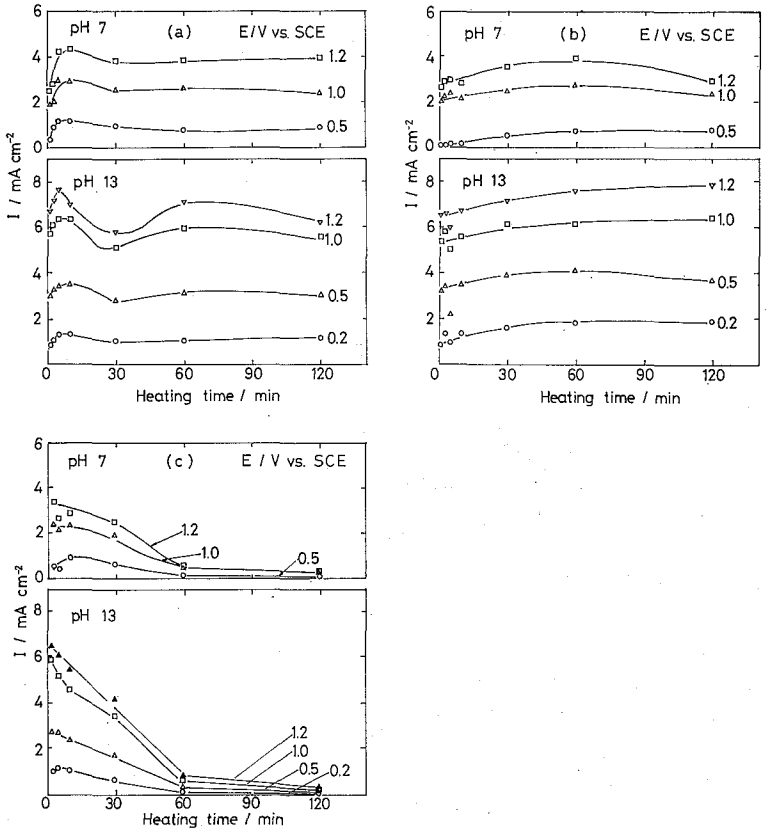

Fig. 8. Heating time dependence of the photo photocurrent in solutions of pH 7 and 13 at severl several bias potentials for Fe_2O_3 film electrodes heated at (a) 600°C, (b) 700°C and (c) 800°C.

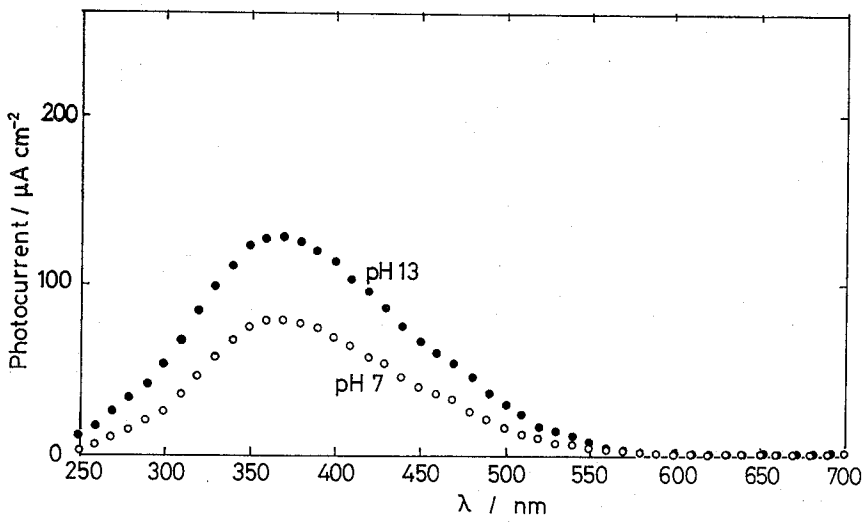

Fig. 9. Action spectra of Fe_2O_3 film electrode heated at 700°C for 5 min.

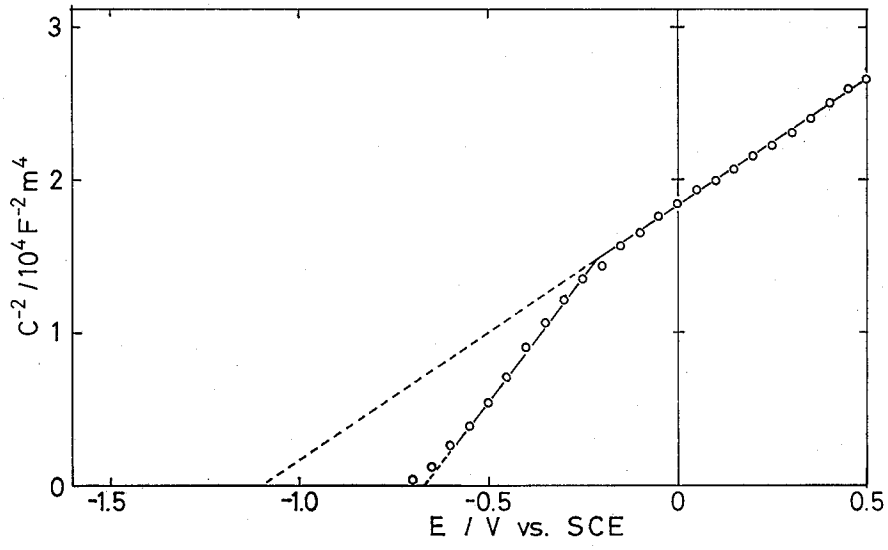

Fig. 10. Mott-Schottky plot of Fe_2O_3 film electode heated at 700°C for 30 min.

In Fig. 10, the Mott-Schottky plot obtained by a.c. impedance analysis is shown for the sample heated at 700°C for 30 min. There is a sharp break around −0.3V (vs. SCE), giving rise to two apparent flat band potentials, −0.67 and −1.09V (vs. SCE).

DISCUSSION

It has been found that the post-heating treatment of the as-prepared Fe_2O_3 films influences markedly the photoelectrochemical properties, especially photocur-

rent as seen from Figs. 5–8. That is, the onset potentials shift towards cathodic direction and the dark currents decrease apparently with increasing heating time at any temperatures. This is considered to be primarly due to the improvement of crystallinity as expected from Fig. 4, although the crystallites cease to grow at a certain time at any heating temperatures.

The heating time dependence of the photocurrent does not seem simple, but basically can be explained by the improvement of crystallinity except heating at 800°C. A sharp decrease in the photocurrent of the samples heated at 800°C with time probably results from the high resistance layer forming between Fe_2O_3 and SnO_2 films, because a large increase in resistance was observed for these samples while there is no appreciable difference in donor density.

Fe_2O_3 electrodes have been known not to show a saturated photocurrent even at deep anodic bias potential, but to show a large dark current and a pronounced transient photocurrent. This is also the case for the present samples derived from sol-gel method. Therefore, this phenomena can be said to be inherent in Fe_2O_3 itself irrespective of the sample preparation processes. They reflect the strong recombination effects not only at the surface but also in the bulk of the materials. The surface recombination centers were found to be diminished to a considerable extent by adding chelating-type supporting electrolyte, resulting in the enhancement of efficiency[7,9]. However, as far as pure Fe_2O_3 is concerned, it may be very difficult to avoid the problems relating to the bulk recombination.

The photocurrent response was observed in the wavelength region below 600

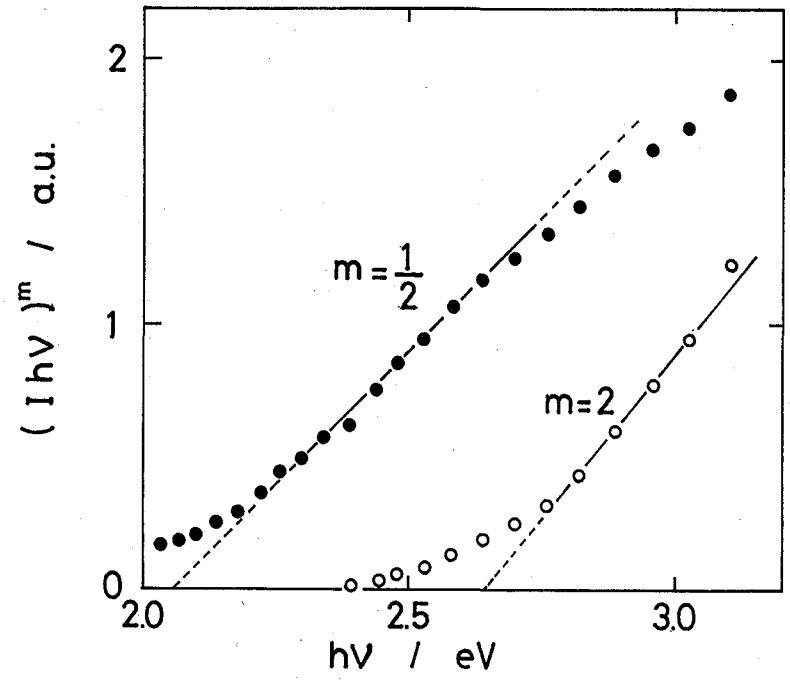

Fig. 11. Plots of $(Ih\nu)^m$ versus $h\nu$ for Fe_2O_3 film electrode heated at 700°C for 5 min.

nm as cleary seen from Fig. 9. It is interesting to note again that a smaller maximum appeared at 475 nm in the action spectra. This suggests that two different transitions are involved in the excitation process. The energy band gap E_g of the interband transition can be determined from the following relation[18],

$$(Ih\nu)^m \propto A(h\nu - E_g)$$

where I is the photocurrent, $h\nu$ the photon energy of an incident light and A the constant depending on the bias potential. For a direct band gap semiconductor, m=2 and for an indirect band gap one m=1/2.

Relations between $(Ih\nu)^m$ and $h\nu$ are shown in Fig. 11. Two straight lines are obtained for m=1/2 in the lower energy region and for m=2 in the higher energy region. From their intercepts to the abscissa, the energies are estimated as 2.06 and 2.64 eV for indirect and direct band gaps, respectively. The indirect band gap energy of 2.06 eV agrees very well with the value of 2.1 eV obtained from the absorption edge in Fig. 2. This result indicates that the smaller band gap is possibly assigned to the indirect $Fe^{3+}(3d^5$ non-bonding)-conduction band (CB) transition. However, as Schumacher *et al.* pointed out[15], the holes generated in the d band have a very low mobility due to its narrowness, leading to their poor transport, in other words, lower conversion efficiency. On the contrary, the larger band gap of 2.64 eV may be assigned to the direct charge transfer due to O (2p bonding)-CB transition. Taking into account that this transition is a major cause of the photocurrent, Fe_2O_3 should be taken as a semiconductor with a band gap energy of 2.64 eV.

Another interesting feature of the Fe_2O_3 film electrodes is to show a sharp break in the Mott-Schottky plot as shown in Fig. 10. Since it was also observed for different kinds of Fe_2O_3 electrodes prepared by sintering[6], and reactive sputtering and plasma oxidation[15], this phenomenon is a intrinsic property of Fe_2O_3. Kennedy

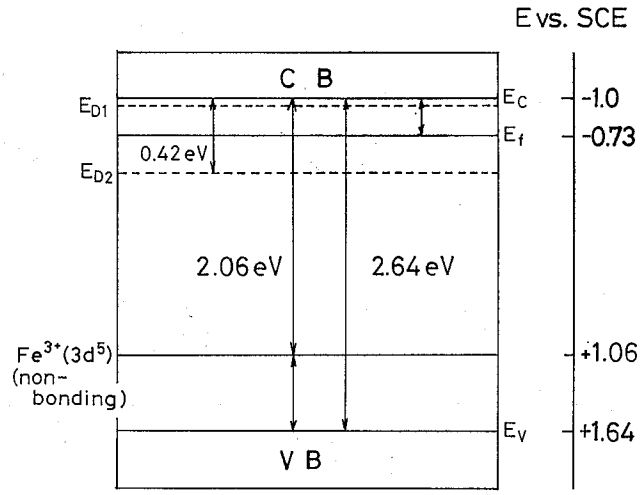

Fig. 12. Energy diagram for the present α-Fe_2O_2 film.

and Frese[6] explained this by assuming the presence of two kinds of donors, shallow and deep. In the present case (Fig. 10), the energy level of the deep donor E_{D2} is estimated as about 0.42 eV below the shallow one E_{D1} from the potential intercepts. The energy diagram for the present α-Fe_2O_3 films can be summarized as in Fig. 12, taking into account the two optical band gaps obtained here. The origin of the shallow donor is possibly assigned to the d electrons in the anti-bonding orbital[6]. Although the deep donor was assigned to Fe^{2+} by Kennedy and Frese[6], the cause is not clear at present.

SUMMARY

Fe_2O_3 has an apparent optical band gap of 2.06 eV which is assigned to the Fe^{3+} (3d non-bonding)-CB transition. However, the photocurrent caused by this transition is very small compared with that due to the second transition from O^{2-} (2p bonding)-CB with a band gap of 2.64 eV. In this respect, Fe_2O_3 is a semiconductor with a band gap of 2.64 eV, rather 2.06 eV.

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