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Electrodeposition and Characterization of γ-FeOOH Thin Films from Oxygen-Bubbled Aqueous Iron Sulfate Solutions

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1. Introduction Iron oxides (e.g. α-Fe_2O_3) and iron oxide hydroxides (e.g. γ-FeOOH) are reported to be suitable candidate materials for wide range of applications. For instance, they absorb visible light, which make them potential materials for photocatalytic and water splitting applications. Additionally, γ-FeOOH can effectively remove heavy metal ions present in contaminated solutions. Interestingly, iron oxide hydroxides can be utilized as starting precursors to produce iron oxides by thermal annealing process. So far, compared to α-Fe_2O_3, no studies have been performed to elucidate the semiconductor properties of γ-FeOOH. Hence, this work focuses on electrodeposition and characterization of γ-FeOOH from aerated aqueous solution containing ferrous ions (Fe^{2+}). Properties such as conductivity-type, photoresponse, optical transmission and band gap are presented and evaluated.

2. Experimental γ-FeOOH thin films were electrodeposited onto tin-doped indium oxide (ITO) coated glass substrate. The deposition was performed in a three-electrode electrochemical cell with ITO substrate, platinum sheet, and saturated calomel electrode (SCE) as working, counter, and reference electrodes, respectively. The solution contained 50 mM $FeSO_4.7H_2O$ and 100 mM KCl. Prior to deposition, the solution was saturated with oxygen by bubbling through the solution. Deposition potentials from -0.7 to -0.9 V were applied for 20 min to electrodeposit the films. All depositions were carried out at room temperature, normal pH (~5.2) and under quiescent solution conditions.

3. Results and Discussion The as-prepared thin films adhered well on the ITO substrate. The thickness of the films ranges from 0.10 - 0.25 μm depending on deposition potential. Figure 1 shows the Raman spectrum of as-prepared sample deposited at -0.9 V. As seen in this figure, two sharp peaks (at 252 and 384 cm^{-1}) and broad and weak peaks (at 538 and 664 cm^{-1}) were observed. These peaks are characteristics Raman shift of γ-FeOOH. Similar Raman peaks were attained for samples deposited at other potentials. From Auger analysis, the estimated Fe/O ratio ranges from 0.52 to 0.54. The ratios agree well with the Raman results. Figure 2 depicts the corresponding SEM photograph. The surface consists of non-uniform aggregates on the background film. Same morphology as seen in Fig. 2 was obtained at -0.8 and -0.7 V. Photosensitivity and conductivity of the as-deposited γ-FeOOH thin film was evaluated from photoelectrochemical (PEC) measurement by employing linearly increasing bias. As shown in Fig. 3, the electrodeposited γ-FeOOH exhibited n-type conductivity and the photoresponse was confirmed, that is, the observed current increased under illumination. Additionally, the estimated band gap is about 2.2~2.6 eV from optical transmission results. The above findings may help in our search of low-cost materials for device applications.

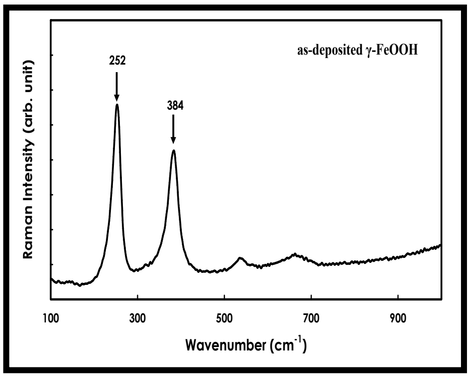

Fig. 1 Raman shift of the as-deposited γ-FeOOH thin film.

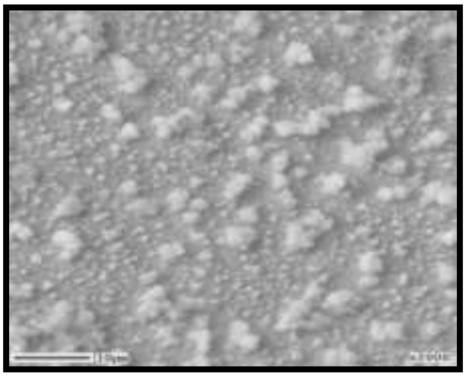

Fig. 2. SEM photograph of the as-prepared γ-FeOOH thin film deposited at -0.9 V

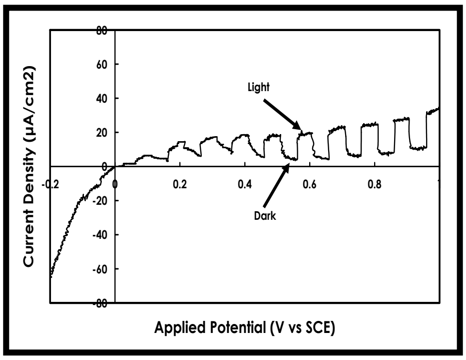

Fig. 3. Photosensitivity of γ-FeOOH film.