Data Article

Optical and electrical features of semitransparent CuO photoelectrochemical cell

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\textbf{A B S T R A C T}

The data presented in this article are related to the research article entitled “CuO photocathode-embedded semitransparent photoelectrochemical cell” (Patel et al., 2016) \cite{1}. This article describes the growth of Cu oxides films using reactive sputtering and application of CuO photocathode in semitransparent photoelectrochemical cell (PEC). In this data article, physical, optical and electrical properties, and PEC performances data set of the reactively sputtered semitransparent CuO samples are made publicly available to enable extended analyses.

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\textbf{Specifications table}

\begin{tabular}{ll}
\textbf{Subject area} & Materials Engineering, Physics, Electrochemistry  \\
\textbf{More specific subject area} & Solar Energy  \\
\textbf{Type of data} & Figures, Table  \\
\textbf{How data was acquired} & Field emission scanning electron microscope (FESEM; JSM-7800F, JEOL Ltd., Tokyo, Japan) Surface profiler (Dektak XT-E, Veeco, Plainview, New York)  \\
\end{tabular}

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UV-visible spectrophotometer (UV-2600, Shimadzu Corporation, Seoul, South Korea).
Potentiostat/Galvanostat (ZIVE SP1, WonA Tech, Korea)
PEC cells (Copper oxide-coated FTO, Ag/AgCl, and platinum gauze were connected to the working, reference, and counter electrodes of the PG-stat, respectively. Aqueous 0.1 M NaOH solution was used as electrolyte)

Data format

Experimental factors
Prepared CuO samples were treated under rapid thermal processing to observe the morphologies before and after air annealing
Surface profiler: contact mode, scanning length 2 mm, and force 5 mg.
Optical Reflectance: CuO photocathode on glass substrate
Mott–Schottky: Frequency→500 Hz to 5 kHz
Bias range→0.4 V to −0.6 V vs. Ag/AgCl

Experimental features
Phase and stoichiometry tunable growth of CuO samples using the reactive sputtering of Cu target, and application in semitransparent photocathode

Data source location
Incheon National University, Incheon-406772, Korea

Data accessibility
The data are with this article

Value of the data

- Performance comparisons of semitransparent CuO photocathode to other Cu oxides (CuOx) based materials. This comparison includes photoelectrochemical (PEC) cell measurement and performance parameters, such as band gaps of CuO materials, types of the electrolyte, light sources, photocurrent density, and methods of CuO fabrication. Readers can easily summarize the progress of CuO PEC cells.
- Approaches to modulate the morphologies of CuOx films. A simple and powerful reactive sputtering method can be applied to tune the CuOx films. The surface morphology of various Cu oxides was obtained by changing the oxygen flow rate during the sputtering process.
- RTP effect is significant and effective to enhance the crystallinity of CuOx films.
- The Mott–Schottky analysis confirms the modulation of built-in potential of CuOx films.
- Tuning of the optical band gap of semitransparent CuO photocathode would be useful for bandgap engineering and applied for advanced CuO embedded photoelectrochemical cells.

1. Data

The comparison for the reactive-sputtered semitransparent CuO photocathode was presented in Table 1. This summary is presented in the chronological order. The readers can easily overview the progress of Cu oxide-based PEC cells. Fig. 1 shows the photographs of the reactive sputtered CuOx films and images after RTP. The surface morphologies of the various Cu oxides grew by various oxygen flows and RTP treatments are presented in Fig. 2. The film thickness measured using the surface profiler is presented in Fig. 3. Reflectance characteristics and Tauc plots of CuO samples are presented in Figs. 4 and 5, respectively. Frequency dependent Mott–Schottky measurement of RTP-treated CuO samples is presented in Fig. 6. Fig. 7 shows the free carrier concentrations and flat band potential of CuO samples according to the Mott–Schottky analysis.
Table 1
Performance comparison for our nanoscaled CuO photocathode with the CuO based photocathodes in literature. Reference potential for measured photocurrent density is mentioned as reversible hydrogen electrode (RHE), silver/silver chloride (Ag/AgCl) and saturated calomel electrode (SCE). Photocurrent value given in this work is the average value of total 3 electrodes.

| Method of preparing CuO | $E_g$ (eV) | Electrolyte | Light source | Photocurrent density (mA cm$^{-2}$) | Year/Ref. |
|-------------------------|------------|-------------|--------------|-------------------------------------|-----------|
| Reactive DC sputtering, room temperature | 1.85 | 0.1 M NaOH | LED, 100 mW cm$^{-2}$ | 1.75 (0.3 V vs. RHE) | This work |
| Reactive DC sputtered + RTP | 1.7 | 0.1 M NaOH | LED, 100 mW cm$^{-2}$ | 6.4 (0.3 V vs. RHE) | This work |
| Grind powder + LiNO$_3$ | 1.35 | 0.1 M Na$_2$HPO$_4$ | Xenon lamp, 810 mW cm$^{-2}$ | ~ 0.44 (~ 0.4 V vs. SCE) | 1982[[2]] |
| Electrodeposition | 1.56 | - | 500 W xenon lamp | ~ 0.08 (~ 0.2 V vs. Ag/AgCl) | 2004[[3]] |
| Sol–gel | 1.77 | NaOH (pH 13) | 150 W xenon arc lamp | ~ 2.02 (~ 0.5 V vs. SCE) | 2009[[4]] |
| Electrochemical two stage growth | - | NaOH (pH 11) | W-halogen lamp, 125 mW cm$^{-2}$ | ~ 0.35 (0.05 V vs. RHE) | 2010[[5]] |
| Spin coating of CuO particle prepared by flame spray pyrolysis | 1.44 | 1 M KOH (pH 14) | 1 sun | 1.2 (~ 0.55 V vs. Ag/AgCl) | 2011[[6]] |
| RF sputtering of CuO | - | 1 M KOH (pH 14) | 150 W solar simulator | ~ 3.15 (~ 0.55 V vs. Ag/AgCl) | 2012[[7]] |
| Flame spray pyrolysis Li:CuO | - | 1 M KOH | 1 sun | ~ 1.69 (~ 0.55 V vs. Ag/AgCl) | 2012[[8]] |
| spinning disk reaction/spin coating | 1.68 | 1 M KOH | 1 sun | 1.58 (~ 0.55 V vs. Ag/AgCl) | 2012[[9]] |
| Solution processed porous CuO | 1.35 | 1 M KOH | 1 sun | 1.2 (~ 0.55 V vs. Ag/AgCl) | 2012[[10]] |
| RF co-sputtered Cu and Ti for Ti:CuO | 1.12–1.46 | 1 M Na$_2$SO$_4$ | 250-W quartz tungsten lamp | 0.09 (~ 0.5 V vs. Ag/AgCl) | 2012[[11]] |
| Sol–gel | 1.2 | 0.1 M Na$_2$SO$_4$ (pH 5.84) | 150 W Xenon arc lamp and AM1.5 filter | ~ 3.5 (~ 0.55 V vs. RHE) | 2014[[12]] |
| Doped CuO by flame spray pyrolysis | - | 1 M KOH (pH 14) | 1 sun | ~ 1.07 (~ 0.55 V vs. Ag/AgCl) | 2014[[13]] |
| Anodising Cu foil: TiO$_2$/CuO | - | 0.5 M K$_2$SO$_4$ | 300 W xenon arc lamp | 2.4 (~ 0.36 V vs. Ag/AgCl) | 2015[[14]] |
| Template assisted electrodeposition of CuO/ZnO | 1.5 | 0.1 M KOH | White light | 1.2 (~ 0.5 V vs. Ag/AgCl) | 2016[[15]] |
| RF sputtering of CuO target | 1.25 | 0.1 M Na$_2$SO$_4$ (pH 5.84) | 1 sun | 2.5 (0 V vs. RHE) | 2016[[16]] |
| RF sputtered CuO + RTP | 1.35 | 0.1 M Na$_2$SO$_4$ (pH 5.84) | 1 sun | 1.68 (0 V vs. RHE) | 2016[[17]] |
| Doped Ni:CuO by flame spray pyrolysis | - | 1 M KOH (pH 14) | 1 sun | 1.07 (~ 0.55 V vs. Ag/AgCl) | 2016[[18]] |
| Spray pyrolysis + Calcination | 1.57 | 1 M KOH (pH 13.5) | 1 sun | 24 (0.25 V vs. RHE) | 2016[[19]] |
| Chemical bath deposition + Calcination | 1.55 | 0.5 M Na$_2$SO$_4$ (pH 6.6) | 1 sun | 1.3 (0 V vs. RHE) | 2017[[20]] |
2. Experimental design, materials and methods

2.1. Preparation of CuO$_x$ films

Large scale (Ø4 in.) Cu target (purity 99.99%) was reactively sputtered to form various phases of copper oxides (CuO$_x$) at room temperature. The reactive gas (O$_2$) and the sputtering gas (Ar) were simultaneously supplied to tune the phases CuO$_x$ by changing the O$_2$ flow rate (1–9 sccm) at a fixed Ar supply (30 sccm). Three types of CuO$_x$ phases were achieved for Batch-1 (Ar/O$_2$ of 30/1 sccm), Batch-3 (Ar/O$_2$ of 30/5 sccm), and Batch-5 (Ar/O$_2$ of 30/9 sccm). To control the film properties, rapid thermal process was performed for 10 min at 550°C. The RTP-treated samples were denoted as Batch-2 (RTP-treated Batch-1), Batch-4 (RTP-treated Batch-3), and Batch-6 (RTP-treated Batch-5), respectively [1]. In order to remain a pure FTO, the Krypton tape was partially covered the FTO glass and removed after the reactive sputtering process.

2.2. PEC Mott–Schottky measurements

The potentiostat/galvanostat (PG-stat; ZIVE SP1, WonA Tech, Seoul, South Korea) was applied for the PEC Mott–Schottky measurements in a three electrodes cell. Copper oxide-coated FTO, Ag/AgCl, and platinum gauze were connected to the working, reference, and counter electrodes of the PG-stat, respectively. Aqueous 0.1M NaOH solution was used as an electrolyte for all PEC measurements.

The measured potential $V$ vs. Ag/AgCl was converted to the reversible hydrogen electrode (RHE) scale according to the Nernst equation: $E_{\text{RHE}} = E_{\text{Ag/AgCl}} + 0.059 \, \text{pH} + E_{\text{Ag/AgCl}}$, where $E_{\text{RHE}}$ is the converted potential vs. RHE, $E_{\text{Ag/AgCl}} = 0.1976 \, \text{V}$ at 25°C, and $E_{\text{Ag/AgCl}}$ is the experimentally measured potential against Ag/AgCl reference. The Mott–Schottky (1/$C^2$ vs. $V$) analysis of photoelectrodes was performed in varying the frequencies (500 Hz to 5 kHz) with the AC signal of 10 mV.
Fig. 2. SEM images of the samples featuring the surface morphological variation for given synthesis condition. Left images presents as sputtered samples, the surface morphology of various Cu oxides prepared by changing the oxygen flow rate. Right images presents samples treated by atmospheric RTP, the surface morphology of nanoscaled CuO converted from various Cu oxides. Scale bar, 1 μm.
Fig. 3. Depth profiles of batch samples. The estimated thin film thickness, process parameters of the samples are marked in each plot.

Fig. 4. Reflectance profiles of as sputtered and RTP treated samples.
Fig. 5. Tauc plot of RTP-treated samples.

Fig. 6. Mott–Schottky plots of samples treated by RTP (a) Batch 2, (b) Batch 4 and (c) Batch 6. These samples present various nanoscale features of CuO materials. Here, $1/C^2$ vs. $V$ shown for various frequencies from 500 Hz to 5 kHz. Consistence slope and intersection on potential axis firm the accurate accepter carrier concentration and flat band potentials of these samples are attributed to the bulk properties and without involving surface states.

Fig. 7. Estimated accepter carrier concentration ($N_A$) and flat band potential ($V_{FB}$) from Mott–Schottky analysis of the samples treated by RTP.
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Transparency document. Supplementary material

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.dib.2018.01.074.

References

[1] M. Patel, H. Kim, D.B. Patel, J. Kim, CuO photocathode-embedded semitransparent photoelectrochemical cell, J. Mater. Res. 31 (2016) 3205–3213.
[2] F.P. Koffyberg, F.A. Benko, A photoelectrochemical determination of the position of the conduction and valence band edges of p-type CuO, J. Appl. Phys. 53 (1982) 1173–1177.
[3] K. Nakaoa, J. Ueyama, K. Ogura, Photoelectrochemical behavior of electrodeposited CuO and Cu2O thin films on conducting substrates, J. Electrochem. Soc. 151 (2004) C661–C665.
[4] D. Chauhan, V.R. Satsangi, R. Shrivastav, Preparation and characterization of nanostructured ZnO thin films for photoelectrochemical splitting of water, Bull. Mater. Sci. 29 (2009) 709–716.
[5] L. Chen, S. Shet, H. Tang, H. Wang, T. Deutsch, Y. Yan, J. Turner, M. Al-Jassim, Electrochemical deposition of copper oxide nanowires for photoelectrochemical applications, J. Mater. Chem. 20 (2010) 6962–6967.
[6] C.-Y. Chiang, K. Aroh, S.H. Ehrman, Copper oxide nanoparticle made by flame spray pyrolysis for photoelectrochemical water splitting – Part II. Photoelectrochemical study, Int. J. Hydrog. Energy 37 (2011) 4871–4879.
[7] C.-Y. Chiang, J. Epstein, A. Brown, J.N. Munday, J. Culver, S.H. Ehrman, Biological templates for antireflective current collectors for photoelectrochemical cell applications, Nano Lett. 12 (2012) 6005–6011.
[8] C.-Y. Chiang, Y. Shin, S. Ehrman, Li doped CuO film electrodes for photoelectrochemical cells, J. Electrochem. Soc. 159 (2012) B227–B231.
[9] C.Y. Chiang, M.H. Chang, H.S. Liu, C.Y. Tai, S. Ehrman, Process intensification in the production of photocatalysts for solar hydrogen generation, Ind. Eng. Chem. Res. 51 (2012) 5207–5215.
[10] C.Y. Chiang, Y. Shin, K. Aroh, S. Ehrman, Copper oxide photocathodes prepared by a solution based process, Int. J. Hydrog. Energy 37 (2012) 8232–8239.
[11] H. Tang, M.A. Matin, H. Wang, S. Sudhakar, L. Chen, M.M. Al-Jassim, Y. Yan, Enhancing the stability of CuO thin-film photoelectrodes by Ti alloying, J. Electron. Mater. 41 (2012) 3062–3067.
[12] Y.-F. Lim, C.S. Chua, C.J.J. Lee, D. Chi, Sol-gel deposited Cu2O and CuO thin films for photocatalytic water splitting, Phys. Chem. Phys. 16 (2014) 25928–25934.
[13] C.-Y. Chiang, Y. Shin, S. Ehrman, Dopant effects on copper oxide photoelectrochemical cell water splitting, Energy Procedia 61 (2014) 1799–1802.
[14] P. Wang, X. Wen, R. Amal, Y.H. Ng, Introducing a protective interlayer of TiO2 in Cu2O–CuO heterojunction thin film as a highly stable visible light photocathode, RSC Adv. 5 (2015) 5231–5236.
[15] U. Shaislamov, K. Krishnamoorthy, S.J. Kim, A. Abidov, B. Allabergenov, S. Kim, S. Choi, R. Suresh, W.M. Ahmed, H.-J. Lee, Highly stable hierarchical p-CuO/ZnO nanorod/nanobranch photoelectrode for efficient solar energy conversion, Int. J. Hydrog. Energy 41 (2016) 2253–2262.
[16] S. Masudy-Panah, R. Siavash Moakhar, C.S. Chua, H.R. Tan, T.L. Wong, D. Chi, G.K. Dalapati, Nanocrystal engineering of sputter-grown CuO photocathode for visible-light-driven electrochemical water splitting, ACS Appl. Mater. Interfaces 8 (2016) 1206–1213.
[17] S. Masudy-Panah, R.S. Moakhar, C.S. Chua, A. Kushwaha, T.I. Wong, G.K. Dalapati, Rapid thermal annealing assisted stability and efficiency enhancement in a sputter deposited CuO photocathode, RSC Adv. 6 (2016) 29383–29390.
[18] C.Y. Chiang, Y. Shin, S. Ehrman, Dopant effects on conductivity in copper oxide photoelectrochemical cells, Appl. Energy 164 (2016) 1039–1042.
[19] M. Patel, R. Pati, P. Marathey, J. Kim, I. Mukhopadhyay, A. Ray, Highly photoactive and photo-stable spray pyrolyzed teneirite CuO thin films for photoelectrochemical energy conversion, J. Electrochem. Soc. 163 (2016) H1195–H1203.
[20] A. Ray, I. Mukhopadhyay, R. Pati, Y. Hattori, U. Prakash, Y. Ishii, S. Kawasaki, Optimization of photoelectrochemical performance in chemical bath deposited nanostructured CuO, J. Alloy. Compd. 695 (2017) 3655–3665.