Photoelectrochemical Properties of Fe$_2$O$_3$–Cu$_2$O Heterojunction Nanowire Prepared on Anodic Aluminum Oxide Template by Sequential Electrodeposition

Received October 20, 2020; revised November 16, 2020; accepted November 20, 2020

Myung Jong Kang$^a$ and Chang Woo Kim$^{b,c,*}$

$^a$Department of Chemistry, Gangneung-Wonju National University, Gangneung 25457, Republic of Korea
$^b$Department of Smart Green Technology Engineering, Pukyong National University, Busan 48513, Republic of Korea
$^c$Department of Graphic Arts Information Engineering, Pukyong National University, Busan 48513, Republic of Korea

*Corresponding author E-mail: kimcw@pknu.ac.kr

ABSTRACT

Fe$_2$O$_3$–Cu$_2$O heterojunction nanowire arrays are a promising photoelectrode technology for solar fuel production because of their increased absorption ability as well as the enhanced separation and transfer rate of the charge carriers. Here, we report on the preparation of a Fe$_2$O$_3$–Cu$_2$O heterojunction nanowire photoelectrode by sequential electrodeposition and we investigate its photoelectrochemical properties. The Fe$_2$O$_3$–Cu$_2$O heterojunction photoelectrode is successfully prepared using an anodic aluminum oxide (AAO) membrane as a template material. One side of a double-pass AAO membrane is covered with conductive Ag paste and then Fe and Cu are sequentially electrodeposited in the metal salt electrolyte for constructing the heterojunction nanowire arrays. The sequentially deposited Fe–Cu nanowire arrays are converted into Fe$_2$O$_3$–Cu$_2$O nanowire photoelectrodes by a post-annealing process. The front side of the crystallized Fe$_2$O$_3$–Cu$_2$O heterojunction nanowire photoelectrode undergoes a photoelectrochemical reaction upon illumination with solar light, and its improved photoelectrochemical properties are investigated intensively. In summary, we propose a facile electrodeposition method to prepare a p–n heterojunction electrode with high charge separation efficiency and improved charge transport efficiency for artificial photosynthesis through solar water splitting and the hydrogen evolution reaction.

Keywords: Heterojunction, Photoelectrode, Sequential deposition

1. Introduction

Thus far, solar energy has been widely studied as a desirable source of renewable and sustainable energy [1–3]. Among various approaches for utilizing solar energy, artificial photosynthesis, which imitates the process of natural photosynthesis, has been highlighted as a promising strategy for producing valuable chemicals such as hydrogen, formic acid, and alcohols [4,5]. In recent decades, the development of the solar-energy-driven water splitting reaction, which splits water molecules into hydrogen and oxygen through a photoelectrochemical process, has been intensively studied because water splitting by solar energy is a basic reaction for realizing an efficient artificial photosynthetic system [6,7]. $\text{H}_2$ (hydrogen), which is a storable and transportable fuel and feedstock, is an ecofriendly resource in the modern chemical industry.

Since Fujishima and Honda discovered the solar-energy-driven water splitting reaction using TiO$_2$ as a photocatalyst through photochemical (PC) and photoelectrochemical (PEC) processes, various types of metal oxide materials such as Fe$_2$O$_3$, BiVO$_4$, ZnO, and WO$_3$ have been used to produce hydrogen through water splitting [8–11]. Strategies for achieving efficient solar energy conversion have focused on three fundamental processes: light absorption, charge separation, and charge transport. Valence band potential engineering, introducing a dopant into a host, using solid solutions, and combining surface plasmon resonance with other technologies have been reported as effective strategies for enhancing physicochemical and photophysical performances. Moreover, together with morphology control of photocatalysts for achieving a large surface area and highly reactive properties, Z-scheme configurations such as composite structures of double-species metal oxide photocatalysts and combining carbon-based materials with photocatalysts have been recognized as prospective strategies [12–14].

Compared with PC systems, morphology structuring from nano- to micro-scale dimensions has attracted considerable attention in the field of PEC systems. One-dimensional (1D) manipulation, such as that of wire-, tube-, and rod-shaped nanomaterials, has been successfully demonstrated to enhance their PEC characteristics. For instance, Yang et al. [15] demonstrated that 1D geometrical parameter manipulation increased the electron transport rate considerably. Furthermore, Frank et al. [16] succeeded in achieving a low recombination rate in 1D structured nanomaterials, which resulted in a high photoconversion...
efficiency. Grimes et al. [17] reported that introducing a 1D TiO$_2$ nanotube into a dye-sensitized solar cell remarkably improved its photovoltaic performance [17]. The above-mentioned results indicate that vertically oriented 1D structured materials exhibit highly improved light energy conversion efficiency with favorable electron and ion diffusion between their interfaces.

However, in spite of the advantages of vertically oriented 1D structured electrodes for solar-energy-driven water splitting in PEC systems, it remains challenging to not only construct a suitable heterojunction of metal-oxide-based photocatalysts with higher chemical stability in a wide range of pH of the electrolyte solution but also maintain the vertically oriented 1D rod/wire structure. Hematite (α-Fe$_2$O$_3$) phase, one of the most abundant minerals on Earth, has high corrosion resistivity in both alkaline and acidic media as well as an optimum band position for high-efficiency solar energy absorption and utilization [18]. Cu$_2$O, which is another abundant mineral, is non-toxic and involves a low-cost fabrication process; hence, it is also capable of good PEC performance [19]. Moreover, it has a band gap of 2.0 eV, which is suitable for high solar energy utilization. Although both Cu$_2$O and hematite have favorable band energy positions for solar water splitting, there are challenges that restrict their application as efficient and durable photoelectrodes for solar fuel production. The major challenges are the unfavorable ratio between the carrier diffusion length and the light absorption depth in the photoelectrode [20,21]. For instance, theoretically, the film thickness of a Cu$_2$O electrode should be at least 1000 nm so that it can absorb sunlight efficiently. However, owing to the short carrier diffusion length of Cu$_2$O (~200 nm or less), it is not possible to increase the thickness of the Cu$_2$O film to the optimal value [22,23]. Hence, it is necessary to construct a p-n junction of a photocatalyst exhibiting much higher photoactivity than bare materials by facilitating the separation and transfer of charge carriers through an internal electric field established at the interface of the heterojunction [24].

To solve the above-mentioned problems, here, we demonstrate the preparation of a Fe$_2$O$_3$–Cu$_2$O heterojunction nanowire photoelectrode by sequential electrodeposition with an anodic aluminum oxide (AAO) membrane as a template material. Through our template-mediated method for constructing a p-n junction structured electrode of hematite and Cu$_2$O with a 1D nanowire structure, we realize high charge separation efficiency and improved charge transport efficiency. In addition, the photoelectrochemical properties for solar fuel production are investigated in detail.

2. Experimental details

2.1. Materials

AAO membranes were purchased from InRedox Ltd. Conductive Ag paste, iron (II) sulfate heptahydrate (FeSO$_4$·7H$_2$O), copper (II) sulfate pentahydrate (CuSO$_4$·5H$_2$O), boric acid (H$_3$BO$_3$), and NaOH were purchased from Sigma-Aldrich. All the chemicals used as received. Distilled water was produced using a Millipore Milli-Q purification system (resistivity $\geq$ 18.2 MΩ·cm).

2.2. Preparation of Fe$_2$O$_3$ and Cu$_2$O nanowire arrays by electrodeposition

Typically, one side of the double-pass AAO wafer was covered with conductive Ag paste. After the conductive Ag paste dried, the AAO membrane was immersed in 0.2 M iron (II) sulfate heptahydrate and 0.1 M boric acid solution as an electrolyte solution. A three-electrode system was configured, where the AAO membrane covered with Ag paste was the working electrode, a coiled platinum wire was the counter electrode, and Ag/AgCl (3 M KCl saturated) was the reference electrode. Electrochemical deposition was performed using a PL-9 (KST-P1) potentiostat at room temperature (RT). Fe wire arrays were electrodeposited at -4 V for 4 h into the AAO column.

After the deposition, the AAO substrate was cleaned with distilled water and ethanol. Then, the AAO substrate was heat-treated at 723 K for 4 h under air conditioning for crystallization. The annealed AAO substrate was immersed in 2 M NaOH solution to remove the AAO membrane. For the preparation of Cu$_2$O nanowire arrays, electrodeposition was performed in 0.2 M copper (II) sulfate and 0.1 M boric acid solution at -4 V for 0.5 h.

2.3. Preparation of Fe$_2$O$_3$–Cu$_2$O nanowire arrays by sequential electrodeposition

Similarly, the electrodeposition of the Fe–Cu metallic bilayers followed the above-mentioned method. Fe wire arrays were deposited on AAO as the first layer under an iron source electrolyte and Cu wire arrays were then sequentially deposited on the Fe wire arrays on AAO under a copper source electrolyte. The sequentially electrodeposited AAO substrate was cleaned and annealed at 723 K for 4 h under air conditioning for the conversion to metal oxide phase crystallization. Finally, it was treated with NaOH to remove the AAO template.

2.4. Characterization

The morphology and elemental mapping of the prepared samples were investigated using a scanning electron microscopy (Hitachi Horiba S300) with attached energy dispersive spectroscopy (EDS) equipment. For transmission electron microscopy (TEM) observations, the samples were prepared by placing one drop of the colloidal solution onto 200-mesh carbon-coated copper grids and dried for a few minutes. The crystallinity and crystal structure were measured by X-ray diffraction (XRD, Rigaku D’Max 2200V) with Cu Ka radiation ($\lambda = 1.5406$ Å). Linear sweep voltammetry (LSV) of each electrode was conducted using a PL-9 (KST-P1) potentiostat with 0.1 M NaOH solution in the three-electrode system. The AAO membrane was the working electrode, a coiled platinum wire was the counter electrode, and Ag/AgCl (3M KCl saturated) was the reference electrode. All the curves were measured under chopped 1-sun illumination (AM 1.5 G, 100 mW/cm$^2$) using a solar simulator (Newport, 300 W Xe source).

3. Results and discussion

In the present study, we demonstrated the preparation of 1D binary wire arrays as a heterojunction photoanode by sequential deposition for solar fuel production. A heterojunction of Fe$_2$O$_3$–Cu$_2$O binary wire arrays was prepared in three steps: 1) binary layer deposition of Fe and Cu wires onto an AAO membrane covered with conductive Ag paste; 2) heat treatment for the conversion of the metal wires into metal oxides, and 3) removal of the AAO membrane by treatment with NaOH. A schematic for the preparation of the Fe$_2$O$_3$–Cu$_2$O binary wire arrays by electrodeposition on the AAO membrane...
AAO nanotube arrays [Fig. 2(b)]. AAO membrane covered with Ag conductive paste. First deposition of Fe nanowires onto alumina electrode, second deposition of Cu nanowires, and heat-treated electrode after removing alumina membrane.

Figure 1. Preparation of nanowire arrays using alumina membrane by sequential electrodeposition. (a) Commercial alumina membrane, (b) AAO membrane covered with Ag conductive paste, (c) first deposition of Fe nanowires onto alumina electrode, (d) second deposition of Cu nanowires, and (e) heat-treated electrode after removing alumina membrane.

Figure 2. Field emission SEM (FE-SEM) and elemental mapping images of (a) alumina membrane and (b) Fe\textsubscript{3}O\textsubscript{4}/Cu\textsubscript{2}O heterojunction nanowire on alumina membrane.

is shown in Fig. 1. First, the double-pass AAO membrane was used [Fig. 1(a)]. One side of the AAO membrane was covered with conductive Ag paste for the preparation of the electrode substrate [Fig. 1(b)]. After the conductive Ag paste dried, FeOOH was electrodeposited inside the column of the AAO membrane using FeSO\textsubscript{4} solution in boric acid as the deposition electrolyte [Fig. 1(c)]. The formation of FeOOH is given by \[Fe^{2+} + 2H_2O \rightarrow FeOOH + 3H^+\] [25,26].

As the second wire array, Cu(OH)\textsubscript{2} was sequentially electrodeposited on FeOOH nano-rods in CuSO\textsubscript{4}–boric acid electrolyte solution [Fig. 1(d)]. The formation reaction of Cu\textsubscript{2}O is given by \[2Cu^{2+} + 2e^- + H_2O \rightarrow Cu_2O + 2H^+\] [27].

The morphology of the Fe and Cu precursors deposited into the column of the AAO membrane was investigated by scanning electron microscopy (SEM). Figure 2(a) shows the top-view and cross-sectional SEM images of the bare AAO membrane. It appears that the AAO template has the morphology of typical nano-tubular/column arrays. Compared with the SEM images of the bare AAO template, Fig. 2(b) shows the top-view and cross-sectional SEM images of the sequentially electrodeposited samples. It indicates that the electrodeposited wires filled the column of the AAO substrate. Interestingly, the cross-sectional SEM image with the EDS mapping results shows that the Fe-based wire arrays were grown from the Ag paste substrate on the bottom of the AAO membrane. As the second deposition layer, Cu-based wire arrays were formed on the Fe-based wire arrays inside the AAO nanotube arrays [Fig. 2(b)].

The Fe–Cu nanowire arrays sequentially deposited on the AAO substrate were heat-treated for conversion into Fe\textsubscript{3}O\textsubscript{4}/Cu\textsubscript{2}O nanowire photoelectrodes. During the annealing process, the electrodeposited Fe-based wires were expected to convert into Fe\textsubscript{3}O\textsubscript{4} and the crystallinity of Cu\textsubscript{2}O was expected to increase as follows [28,29]:

\[2FeOOH + H_2O \rightarrow Fe_3O_4\]

To free the binary wire arrays, the Ag-covered AAO substrate was immersed in NaOH solution and then removed. Their wet-etching reaction is expressed as

\[2OH^- + Al_2O_3 \rightarrow 2AlO_2^- + H_2O\]
\[6H^+ + Al_2O_3 \rightarrow 2Al^3+ + 3H_2O\]

As the above-mentioned wet-etching reaction occurs drastically in NaOH solution, the sequentially deposited products were treated with NaOH [Fig. 1(e)]. After dissolving the AAO membrane in NaOH solution, the morphology of the free-standing binary wire arrays was observed via SEM analysis (Fig. 3). Figure 3(a) shows the top-view SEM image of the structured nanowires with a diameter of around 100 nm, where the pore diameter of the AAO nanotube template was maintained after sequential deposition of the nanowire structure. The array of deposited nanowires was also observed with a length of around 5 μm in the cross-sectional SEM image [Fig. 3(b)]. The selected area of the prepared nanowire arrays was observed via EDS mapping analysis [Fig. 3(c)]. The selected area electron diffraction (SAED) pattern of the individual nanowires was compared among three zones of nanowires. The SAED pattern of the first zone [red box in Fig. 4(a)] showed typical crystalline Fe\textsubscript{3}O\textsubscript{4} with the (110) plane lattice structure in which the d-spacing was 0.2521 nm [Fig. 4(b)] [30]. The SAED pattern of the second zone [blue box in Fig. 4(a)] showed typical crystalline Cu\textsubscript{2}O with the (311) plane lattice structure in which the d-spacing was 0.4266 nm [Fig. 4(c)] [31].
Interestingly, the EDS result for the middle zone [green box in Fig. 4(a)] in the individual nanowires shows that both Fe and Cu exist concurrently [Fig. 4(d)]. Together with EDS analysis, the SAED pattern of the middle zone of the nanowires showed typical SAED patterns of a multi-crystal structure, which is different from the SAED pattern of single crystal phase materials. This indicates that the middle part of the heterojunction nanowire consisted of a mixed/combined crystal structure of Cu₂O and Fe₂O₃ phases after the post-annealing process of the heterojunction of Fe₂O₃-Cu₂O binary wire arrays [Fig. 4(e)]. These results are consistent with previous SEM-EDS analysis results in which Cu₂O and Fe₂O₃ exist simultaneously in the middle zone of the Fe₂O₃-Cu₂O heterojunction nanowire arrays.

To determine the effect of the post-annealing process on the conversion into metal oxide phases after sequential electrodeposition, the crystal structures of the nanowire arrays were compared according to different post-annealing treatments with XRD patterns (Fig. 5). When the binary stacked nanowire array was heat-treated at 300 °C, only two peaks was dominantly observed at 2θ values of 25° and 74° [Fig. 5(a)]. They matched with the (012) plane of the Fe₂O₃ phase and the (311) plane of the Cu₂O phase, respectively. As the annealing temperature was increased to 400 °C, hidden peaks of other planes appeared in both the Fe₂O₃ phase and the Cu₂O phase. The observed peaks at 25°, 42°, 50°, and 64° correspond to the (110), (113), (024), and (300) planes of the hematite phase, respectively. Other peaks at 37°, 44°, 62°, and 77° were assigned to the (111), (200), (220), and (222) planes of the Cu₂O phase, respectively. This indicates that the crystallinity of the Fe₂O₃-Cu₂O heterojunction increased with the post-annealing temperature [Fig. 5(c)].

Based on the previously reported band energy position of Cu₂O and Fe₂O₃ as a visible-responsive photocatalyst, the suggested energy band diagram of the Fe₂O₃-Cu₂O heterojunction nanowire is presented as a scheme in Fig. 6(a). Specifically, the band position of the Cu₂O photocatalyst ranges from 1.3 V vs. NHE for the valance band energy level to -0.7 V vs. NHE for the conduction band energy level, while the band position of Fe₂O₃ ranges from 2.4 V vs. NHE for the valance band energy level to 0.3 V vs. NHE for the conduction band energy level [22,32]. Considering the intrinsic properties of these photocatalysts, i.e., Fe₂O₃ has n-type semiconductor properties and Cu₂O has p-type semiconductor properties, constructing a p-n junction of both materials facilitates a more favorable hole-charge separation and diffusion of the generated holes and electrons owing to the inner electric field induced by the p-n junction [33]. This is proved by LSV measurements of the Fe₂O₃-Cu₂O heterojunction nanowire photoelectrode, as shown in Fig. 6(b). The LSV curve of the pure Fe₂O₃ electrode is a typical LSV curve of an n-type semiconductor, with an onset potential (V onset) of 0.45 V vs. Ag/AgCl, and the 1-sun illumination response starts at 0.5 V vs. Ag/AgCl in the anodic direction. The bare Fe₂O₃ photoanode exhibited a current density of 0.16 mA/cm² at 1.23 V vs. Ag/AgCl under the standard reaction potential for the water oxidation reaction. In addition, no light response was observed at a potential lower than 0.5 V vs. Ag/AgCl. Further, pure Cu₂O shows typical LSV curves of a p-type semiconductor, with a V onset of 0.2 V vs. Ag/AgCl, and the 1-sun illumination response starts at 0.2 V vs. Ag/AgCl in the cathodic direction. In addition, the bare Cu₂O photoelectrode showed a current density of 0.1 mA/cm² at -0.3 V vs. Ag/AgCl and no light response was observed at a potential higher than 0.2 V vs. Ag/AgCl. However, interestingly, the Fe₂O₃-Cu₂O nanowire showed 1-sun illumination responses in the whole range of the LSV curves, with an anodic current density of 0.23 mA/cm² at 1.23 V. Compared with that of bare Fe₂O₃, the current density of the Fe₂O₃-Cu₂O heterojunction nanowire was 60 % greater at 1.23 V vs. Ag/AgCl. Moreover, the Fe₂O₃-Cu₂O heterojunction nanowire showed a cathodic current of 0.8 mA/cm² at -0.3 V, which was not observed in the case of the bare Fe₂O₃ photoelectrode. It is worth noting that the effect of the p-n junction facilitates a more favorable hole-charge separation and diffusion of the generated holes and electrons owing to the inner electric field induced by the p-n junction [33].
was obvious in the case of the Fe$_2$O$_3$–Cu$_2$O heterojunction nanowire structure with the shift of the anodic onset potential from 0.45 to 0 V vs. Ag/AgCl. These remarkable enhancements in the photoelectrochemical activity of the Fe$_2$O$_3$–Cu$_2$O nanowire structure were realized by constructing a heterojunction structure consisting of both the photocatalysts. This strongly suggests that their hole–charge separation are more favorable and the charges are readily diffused owing to the p–n junction structure of the Fe$_2$O$_3$–Cu$_2$O binary nanowire.

4. Conclusions

In this study, a facile approach for enhancing the photoelectrochemical properties of Fe$_2$O$_3$ and Cu$_2$O nanowire array photoelectrodes was investigated by constructing a p–n heterojunction structure of both the materials. The Fe$_2$O$_3$–Cu$_2$O heterojunction nanowire was prepared by electrodeposition on an AAO membrane as a template for the photoelectrode. Through a post-annealing process, high crystallinity was achieved for improving the charge transport efficiency of the photoelectrode. As a result, the sequentially deposited binary wire arrays exhibited enhanced PEC performance compared to the individual materials. Moreover, a significant $V_{\text{onset}}$ shift of the Fe$_2$O$_3$–Cu$_2$O heterojunction nanowire array electrode was observed. In summary, we proposed a facile method for preparing a p–n heterojunction electrode by sequential electrodeposition, with high charge separation efficiency and improved charge transport efficiency for artificial photosynthesis through solar water splitting and the hydrogen evolution reaction.

Acknowledgements

This work was supported by a research grant from Pukyong National University (2019).

References

[1] N. S. Lewis and D. G. Nocera, PNAS 103, 15729 (2006).
[2] V. G. Gude, N. Nirmalakhandan, and S. Deng, Energy 36, 78 (2011).
[3] N. S. Lewis, Science 351, aad1920 (2016).
[4] M. J. Kang, C. W. Kim, A. U. Pawar, H. G. Cha, S. Ji, W. B. Cai, and Y. S. Kang, ACS Energy Lett. 4, 1549 (2019).
[5] C. W. Kim, M. J. Kang, S. Ji, and Y. S. Kang, ACS Catal. 8, 968 (2018).
[6] H. W. Seo and J. S. Kim, Appl. Sci. Converg. Technol. 27, 61 (2018).
[7] T. Nakamura, Sol. Energy 19, 467 (1977).
[8] H. S. Kim, Appl. Sci. Converg. Technol. 28, 122 (2019).
[9] S. H. Nam and J. H. Boo, Appl. Sci. Converg. Technol. 22, 138 (2013).
[10] C. W. Kim, S. Ji, M. J. Kang, H. Park, F. Li, H. M. Cheng, and Y. S. Kang, Mater. Today Energy 13, 205 (2019).
[11] A. Fujishima and K. Honda, Nature 238, 37 (1972).
[12] H. G. Cha, H. S. Noh, M. J. Kang, and Y. S. Kang, New J. Chem. 37, 4004 (2013).
[13] J. Low, J. Yu, M. Jaroniec, S. Wageh, and A. A. Al-Ghamdi, Adv. Mater. 29, 1601694 (2017).
[14] C. W. Kim, M. J. Kang, T. K. Van, and Y. S. Kang, J. Ind. Eng. Chem. 53, 341 (2017).
[15] M. Law, L. E. Greene, J. C. Johnson, R. Saykally, and P. Yang, Nat. Mater. 4, 455 (2005).
[16] K. Zhu, N. R. Neale, A. Miedaner, and A. J. Frank, Nano Lett. 7, 69 (2007).
[17] G. K. Mor, K. Shankar, M. Paulose, O. K. Varghese, and C. A. Grimes, Nano Lett. 6, 215 (2006).
[18] K. Sivula, F. Le Formal, and M. Grätzel, ChemSusChem 4, 432 (2011).
[19] J. Luo, L. Steier, M. K. Son, M. Schreier, M. T. Mayer, and M. Grätzel, Nano Lett. 16, 1848 (2016).
[20] A. Paracchino, J. C. Brauer, J. E. Moser, E. Thimsen, and M. Graetzel, J. Phys. Chem. C 116, 7341 (2012).
[21] K. P. Musselman, A. Marin, L. Schmidt-Mende, and J. L. MacManus-Driscoll, Adv. Funct. Mater. 22, 2202 (2012).
[22] A. Paracchino, V. Laporte, K. Sivula, M. Grätzel, and E. Thimsen, Nat. Mater. 10, 456 (2011).
[23] P. E. de Jongh, D. Vanmaekelbergh, and J. J. Kelly, J. Electrochem. Soc. 147, 486 (2000).
[24] M. Yan, Y. Hua, F. Zhu, W. Gu, J. Jiang, H. Shen, and W. Shi, Appl. Catal. B: Environ. 202, 518 (2017).
[25] J. L. Leibenguth and M. Cohen, J. Electrochem. Soc. 119, 987 (1972).
[26] G. Rahman and O. S. Joo, Mater. Chem. Phys. 140, 316 (2013).
[27] G. Wu, W. Zhai, F. Sun, W. Chen, Z. Pan, and W. Li, Mater. Res. Bull. 47, 4026 (2012).
[28] M. J. Kang and Y. S. Kang, J. Mater. Chem. A 3, 15723 (2015).
[29] M. J. Kang, H. Yu, W. Lee, and H. G. Cha, J. Phys. Chem. Sol. 130, 93 (2019).
[30] D. R. Cummins, H. B. Russell, J. B. Jasinski, M. Menon, and M. K. Sunkara, Nano Lett. 13, 2423 (2013).
[31] T. D. Golden, M. G. Shumskey, Y. Zhou, R. A. VanderWerf, R. A. Van Leeuwen, and J. A. Switzer, Chem. Mater. 8, 2499 (1996).
[32] M. Barroso, S. R. Pendlebury, A. J. Cowan, and J. R. Durrant, Chem. Sci. 4, 2724 (2013).
[33] M. Wang, Y. Hu, J. Han, R. Guo, H. Xiong, and Y. Yin, J. Mater. Chem. A 3, 20727 (2015).