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Hydrotalcite-wrapped Co–B alloy with enhanced oxygen evolution activity

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ABSTRACT

Water oxidation is one of the most attractive techniques for intermittent renewable energy conversion and storage. The oxygen evolution electrocatalytic performance of an amorphous Co–B alloy and its derivatives were studied. These materials were chemically synthesized by reducing a Co salt with NaBH₄. The amorphous Co–B alloy showed good electrocatalytic activity in oxygen evolution but its stability was poor. A hydrotalcite-wrapped Co–B alloy was synthesized by mild oxidation. The electrocatalytic activity of this material in the oxygen evolution reaction was better than that of a commercially available Ir/C catalyst.

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1. Introduction

The oxygen evolution reaction (OER) is an important electrocatalytic process in water electrolysis, metal electrowinning, and metal-air batteries. However, the OER has sluggish kinetics, reflected by the need for a large overpotential to obtain a reaction current sufficient for practical applications. The rational design of electrocatalysts that accelerate OER kinetics has been a core activity in OER research in recent years. Most active OER electrocatalysts are based on precious-metal oxides such as RuO₂ and IrO₂, despite their high cost [1]. However, their use in large-scale commercial applications is expensive and challenging because of the cost and stability problems. Catalysts based on low-cost, highly active, non-noble-transition metals are becoming attractive alternatives for use in OERs.

Amorphous materials based on transition-metal compounds are attractive non-noble-metal catalysts because of their specific chemical structures and high concentrations of unsaturat-

ed sites. Amorphous metal-based catalysts have performed well in OERs; for example, amorphous cobalt phosphate showed high activity in neutral water [2]. The OER activities of amorphous transition-metal oxides are superior to those of their crystalline counterparts. Because of their unusual amorphous structures, the OER catalytic properties of the amorphous mixed metal oxides Fe_{100-x-y}Co_yNi_zO_x are comparable to those of certain noble-metal catalysts, and are promising alternative amorphous electrocatalysts to noble-metal catalysts [3]. Furthermore, the surfaces of some crystalline catalysts can be transformed into an amorphous state, resulting in greatly improved OER activities. This was observed for a perovskite catalyst (Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-δ}) in the OER reaction; the results showed that the activity was related to the amorphous surfaces [4].

As well as amorphous metal compounds, amorphous alloys have shown great potential for use in catalytic hydrogenation [5–8] and battery materials [9,10]. Amorphous materials have

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high specific surface areas and amorphous alloys are easily synthesized, therefore their catalytic performance in the OER should be investigated. Transition metal–B alloys were first investigated as OER catalysts [11,12]. In the 1980s, the amorphous alloys $\text{Fe}_{60}\text{Co}_{20}\text{Si}_{10}\text{B}_{10}$ and $\text{Co}_{50}\text{Ni}_{25}\text{Si}_{15}\text{B}_{10}$ were identified as effective electrocatalysts for the OER [13,14]. These alloys were traditionally fabricated using a liquid quenching method, which limits the synthesis of nanostructured catalysts with high electrochemically active surface areas. The OER activities of metal–B alloys are affected by the synthesis temperature. Samples synthesized at medium temperatures are more active because the amorphous phase is converted to a crystalline phase at high temperatures. The industrial methods generally used for synthesizing transition metal–B alloys are (1) reduction of metal oxides using a mixture of boron carbide and carbon, (2) electrolysis, or (3) direct reaction of the elemental precursors [15].

Here, we report the OER performance and structural evolution of a nanostructured Co–B alloy wrapped by hydrotalcite nanosheets. The Co–B alloy was produced by chemically reducing transition-metal ions with NaBH_4 [16]. Hydrotalcite nanosheets were formed synchronously during alloy formation by introducing a weak oxidizing agent. The microstructures and compositions of the Co–B alloy were determined and correlated with the electrochemical OER performance. The results show that wrapping in hydrotalcite nanosheets improved the activity of a slightly oxidized Co–B alloy; its activity was superior to that of a commercially available Ir/C catalyst. The structural evolution during the OER was investigated using X-ray absorption spectroscopy (XAS) and X-ray photoelectron spectroscopy (XPS).

2. Experimental

2.1. Materials

$\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{Co}(\text{Ac})_2 \cdot 4\text{H}_2\text{O}$, NaBH_4 , NaOH , Na_2CO_3 , and ethylene glycol were purchased from Sigma-Aldrich and used as received. All solutions were prepared using deionized water.

2.2. Synthesis

2.2.1. Synthesis of Co–B

In a typical Co–B synthesis, NaBH_4 solution (2 mol/L, 25 mL) was added dropwise to a CoCl_2 solution (0.4 mol/L, 25 mL) under stirring in a nitrogen atmosphere. Before mixing, nitrogen was bubbled through both solutions for 30 min. The precipitated black powder was separated by filtration, washed thoroughly with water, and dried overnight in a vacuum oven at 70 °C.

2.2.2. Synthesis of Co–B–O

In a typical Co–B–O synthesis, 25 mL of an aqueous solution of NaBH_4 (2 mol/L) and NaOH (0.2 mol/L) were added dropwise to a CoCl_2 solution (0.4 mol/L, 25 mL) in air. The precipitated black powder was removed by filtration, washed thoroughly with water, and dried overnight in a vacuum oven at 70 °C.

2.2.3. Synthesis of Co–O

In a typical Co–O synthesis, an aqueous solution of NaOH (0.2 mol/L, 25 mL) was added dropwise to a CoCl_2 solution (0.4 mol/L, 25 mL) in air. The precipitated green powder was removed by filtration, washed thoroughly with water, and dried overnight in a vacuum oven at 70 °C. For comparison, Co_3O_4 was prepared using a previously reported method [17].

2.3. Characterization

The crystal structures of the samples were determined using powder X-ray diffraction (XRD; Bruker D8). XAS was performed using BL16A at the Taiwan National Synchrotron Radiation Research Center. Co K-edge X-ray absorption near-edge structure (XANES) spectroscopy was performed in fluorescence mode using a Lytle detector. The powder samples were adhered to Kapton tape. The incident beam was positioned at 45° to the sample. The spectra were normalized using Athena software. Transmission electron microscopy (TEM) and selected area electron diffraction (SAED) analysis were performed using a FEI F20 transmission electron microscope. The samples for TEM and SAED were prepared by dispersing Co–B in ethanol. XPS was performed using a Kratos Axis ULTRA X-ray photoelectron spectrometer. The incident radiation was monochromatic $\text{Al K}\alpha$ X-rays. The binding energies reported here were corrected using C 1s (284.8 eV) from the carbon contamination layer. Raman spectra were obtained using an Invia Renishaw Raman spectrometer. The excitation source was a 532-nm green laser.

2.4. Electrochemical measurements

Electrochemical measurements were performed using a CHI 900C workstation. For these measurements, a catalyst sample (3 mg) was dispersed in saturated chitosan solution (3 mL) by sonication for 30 min to form a homogeneous ink. For linear sweep voltammetry (LSV) and cyclic voltammetry (CV), the catalyst ink (12 μL) was cast onto the disc (radius 2 mm) of a rotating ring–disc electrode. LSV was performed at 10 mV/s with the working electrode continuously rotating at 1600 r/min. CV was performed at a scanning rate of 30 mV/s. Chronopotentiometry was used to study the structural evolution of the catalyst during the OER. The chronopotentiometric tests were performed at a constant current density of 10 A/g using the catalyst ink (60 μL) cast onto a carbon paste. All tests were performed in 0.1 mol/L KOH solution. A platinum wire was used as the counter electrode and Hg/HgO was used as the reference electrode.

3. Results and discussion

The Co–B sample was synthesized by reducing CoCl_2 with NaBH_4 solution under nitrogen protection. Fig. 1(a) shows the XRD pattern of Co–B; the hump at 53.6° is assigned to an amorphous Co–B alloy. The XANES results for Co–B are shown in Fig. 1(b). The spectrum of the Co–B sample has a prominent shoulder at 7713 eV, which corresponds to Co metal and indi-

Fig. 1. XRD patterns (a) and XANES (b) results for Co-B, Co-B-O, and Co-O.

cates the presence of metallic Co in Co-B. The weak peak at 7727.4 eV is related to Co^{2+} , which may arise from the partial oxidation of cobalt borides on exposure to air [18], and/or cobalt borate formed as a by-product during synthesis [6,19,20].

A Co-B-O sample was produced using air and NaOH as oxidizing agents. The peaks in the XRD pattern of Co-B-O (Fig. 1(a)) are assigned to $\alpha\text{-Co}(\text{OH})_2$ with a large interlayer distance of 0.88 nm. The broadness of the peaks shows that the $\alpha\text{-Co}(\text{OH})_2$ crystallinity is poor. A hump related to the amorphous Co-B alloy is observed at 53.6° . The XANES spectrum of Co-B-O (Fig. 1(b)) shows a shoulder at 7713 eV and a peak at 7727.2 eV. The shoulder is related to metallic Co, which is con-

sistent with the hump in the XRD pattern. The peak at 7727.2 eV is assigned to Co^{2+} , which is associated with the $\alpha\text{-Co}(\text{OH})_2$ phase, as shown by the XRD pattern. The Co^{2+} peak is more prominent than the corresponding peak for the Co-B sample, suggesting the presence of $\alpha\text{-Co}(\text{OH})_2$ in Co-B-O. The XRD and XANES results both suggest that Co-B-O is a mixture of Co-B and $\alpha\text{-Co}(\text{OH})_2$. As a control, $\alpha\text{-Co}(\text{OH})_2$, denoted by Co-O, was synthesized using the same procedure as for Co-B-O, but without adding NaBH_4 . Its XRD pattern in Fig. 1(a) shows a hydrotalcite-like layered structure with an interlayer distance of 0.79 nm. The XANES spectrum of Co-O in Fig. 1(b) shows a strong peak at 7727.4 eV, which is assigned to Co^{2+} .

Fig. 2. TEM images and photographs of Co-B (a), Co-B-O (b), and Co-O (c). Insets show corresponding SAED patterns.

The photographs of the Co-B, Co-B-O, and Co-O samples in Fig. 2 show that they differ in colour and that the Co-B alloys are magnetic. TEM images showing the morphologies of the samples are also shown in Fig. 2. Co-B consists of aggregated globular particles of size 25–50 nm (Fig. 2(a)). These particles have shells of thickness ~ 5 nm (Fig. 2(b)). According to the literature, the shells may consist of amorphous oxides of B and Co, and a cobalt borate phase formed as a by-product during synthesis [6,19,20]. This layer differs from the hydrotalcite nanosheets and has a limited effect on the OER performance. The SAED pattern in the inset shows a diffuse ring corresponding to the amorphous structure. Co-B-O consists of 2–50 nm nanoparticles wrapped by thin sheets (Fig. 2(b)). The area indicated by A in Fig. 2b shows lattice fringes similar to those of Co-O (Fig. 2(c)). The corresponding SAED (left half of the inset) is also similar to that of Co-O, and is assigned to α -Co(OH)₂ based on the XRD results (Fig. 1). However, there are no lattice fringes in area B, and the SAED of this area (right half of the inset) shows a diffused disc that is related to an amorphous structure in the Co-B alloy. Fig. 2(c) shows that Co-O consists of thin sheets, which is the typical hydrotalcite morphology. The SAED pattern also indicates that the Co-O sample is crystalline. These results show that Co-B is mainly amorphous and dominated by the Co-B alloy phase, whereas Co-B-O is a composite consisting of an amorphous alloy wrapped by hydrotalcite nanosheets. The surface compositions of Co-B and Co-B-O were further investigated using XPS. The XPS survey spectra in Fig. 3(a) confirm the presence of oxygen on the surfaces of Co-B and Co-B-O. The high-resolution B 1s and Co 2p XPS spectra in Fig. 3(b) and (c) show the co-existence of cobalt borides, and oxides of Co and B in Co-B. However, cobalt borides were not detected in Co-B-O, indicating that the cobalt borides might have reacted with NaOH to form the hydrotalcite phase.

The OER electrocatalytic activities of the three samples were investigated using voltammetry techniques; the results are shown in Fig. 4. The cyclic voltammogram of Co-B has two primary features: an anodic peak in the potential region 1.15–1.35 V vs the reversible hydrogen electrode (RHE) and an anodic shoulder emerging from 1.55 V, characteristic of the OER process. The first anodic peak corresponds to oxidation of Co²⁺ to Co³⁺. Despite the large amount of metallic Co in Co-B, no

oxidation peak related to the transformation of metallic Co to Co²⁺ is observed because this transformation occurs at 0.1–0.3 V vs the RHE [21]. This suggests that the oxidation current is mainly derived from the exterior cobalt borides in the amorphous shell covering the alloy nanoparticles. The sharp increase in the oxidation current from 1.55 V is attributed to the OER. The Co-B OER overpotential at 10 mA/cm² is smaller than that for Co₃O₄ (Fig. 4(b)). At 1.7 V, the OER current density for Co-B (6.6 mA/cm²) is almost an order of magnitude larger than that for Co₃O₄ (0.85 mA/cm²). Co-B therefore shows considerable electrocatalytic activity in the OER. The CV profiles for Co-B-O and Co-O are similar: both have an anodic peak for the oxidation of hydrotalcite at a potential lower than that for Co-B oxidation. Nevertheless, the anodic current for the oxidation of Co²⁺ to Co³⁺ on Co-B-O is much larger than that on Co-O.

The linear sweep voltammogram of Co-B-O (Fig. 3(b)) also shows an anodic peak related to the oxidation of Co²⁺ to Co³⁺ in the potential region 1.15–1.25 V and an oxidation current attributable to the OER, which starts to increase at 1.52 V. The OER overpotential of Co-B-O is lower than that of Co-B at 10 mA/cm². The OER current density at 1.7 V for Co-B-O is 14.3 mA/cm², more than two-fold greater than that for Co-B. The OER activity of Co-B-O is therefore significantly better than that of Co-B. The OER current density of Co-B-O is even higher than that of the commercially available precious-metal catalyst Ir/C (9.4 mA/cm² at 1.7 V). This improvement is related to the novel structure of Co-B-O, i.e., an α -Co(OH)₂-wrapped Co-B alloy. The effects of α -Co(OH)₂ were investigated by performing LSV on Co-O under the same conditions as for Co-B-O; the results are shown in Fig. 4(b). The OER current for Co-O starts to increase at 1.55 V and reaches 7.2 mA/cm² at 1.7 V. Its activity is slightly higher than that of Co-B, but lower than that of Ir/C. The OER activity of Co-B-O is therefore not simply the sum of the activities of Co-B and α -Co(OH)₂. Phase junctions can significantly improve catalytic activities [22,23]. We postulate that the superior OER activity of Co-B-O is related to phase junctions between the Co-B alloy and α -Co(OH)₂, inducing a synergistic effect.

Chronopotentiometry was performed to determine the stabilities of Co-B and Co-B-O during the OER, and compare them with that of Ir/C (Fig. 4(c)). At a current density of 10 A/g, the Co-B potential increased from 1.6 to 1.76 V during a 12-h test,

Fig. 3. XPS spectra of Co-B, Co-B-O, and Co-O. (a) Survey; (b) B 1s; (c) Co 2p.

Fig. 4. Cyclic voltammograms (a) and linear sweep voltammograms (b) of Co-B, Co-B-O, Co-O, and Co₃O₄, and the current density was calculated based on geometric area of electrode disc; (c) Chronopotentiometric results at constant current density of 10 A/g; (d) Linear sweep voltammograms of Co-B-O and Ir/C electrodes after chronopotentiometric tests.

indicating low stability. In contrast, only a slight potential increase of 20 mV, from 1.58 to 1.6 V, was observed during the chronopotentiometric testing of Co-B-O under the same conditions, indicating excellent stability. In contrast, the Ir/C catalyst started to lose activity within 2 h, as shown by fast polarization. The LSV curves before and after the chronopotentiometric tests are shown in Fig. 4(d). The Co-B-O activity was still good after the stability test, whereas Ir/C was virtually inactive in the OER.

The structural changes in Co-B-O after the OER were clari-

fied using a combination of XANES and XPS. The XANES spectrum of Co-B-O after the OER (Fig. 5(a)) shows that compared with that of the as-prepared Co-B-O, the shoulder assigned to metallic Co shifts to lower energy and the absorption edge shifts to a position close to that for CoOOH, implying that α -Co(OH)₂ present in Co-B-O is oxidized to Co³⁺-containing species during oxidation. The high-resolution Co 2p XPS spectrum in Fig. 5(b) confirms that Co²⁺ on the surface of Co-B-O was oxidized to Co³⁺ during the OER. Fig. 6 shows that the Raman spectrum of the spent Co-B-O is similar to that of

Fig. 5. XANES (a) and XPS (b) results for Co-B-O after chronopotentiometric testing for 12 h.

Fig. 6. Raman spectra of Co-B-O and α -Co(OH)₂ before and after chronopotentiometric testing for 12 h.

α -Co(OH)₂ after the OER, implying that Co-B-O is transformed to amorphous (hydrous) cobalt oxides during the OER; this amorphous phase probably contributes to the superior OER activity and stability of Co-B-O.

4. Conclusions

Amorphous Co alloy-based catalysts are promising candidates for OER electrocatalysts. A novel catalyst consisting of a Co-B alloy wrapped by α -Co(OH)₂ showed greatly improved OER activity and stability, even higher than those of the commercially available precious-metal catalyst Ir/C. At a potential of 1.7 V, the OER current density of the hydrotalcite-wrapped alloy was 14.3 mA/cm², compared with 9.4 mA/cm² for the Ir/C catalyst. XANES and XPS analyses both showed that the Co

atoms on the surface of the composite catalyst were oxidized to Co³⁺, i.e., a chemical coordination environment similar to that of cobalt oxyhydroxide.

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Graphical Abstract

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A combination of amorphous particles and layered sheets can improve water oxidation reactions for clean energy sources.

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层状水滑石结构包覆钴硼合金的电化学产氧性能

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摘要: 电解水制氧是一种非常有吸引力的转化储存间歇式能源的技术. 本文着重研究了非晶态钴硼合金的电解水产氧性能. 非晶态钴硼合金可以通过硼氢化钠直接还原钴盐来获取, 并表现出较高的电化学活性. 对钴硼合金的弱氧化, 产生了类水滑石结构的钴氢氧化物, 可进一步提高该类催化剂的整体催化活性, 达到接近商业催化剂水平.

关键词: 非晶态合金; 产氧反应; 电催化剂

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