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## Short Communication

# Cobalt phosphide nanoparticles film growth on carbon cloth: A high-performance cathode for electrochemical hydrogen evolution



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## ABSTRACT

In this communication, cobalt phosphide nanoparticles film was developed on carbon cloth (CoP NPs/CC) through low-temperature phosphidation of its corresponding Co NPs/CC precursor. When directly used as a cathode for electrochemical hydrogen evolution in strongly acidic solutions, the CoP NPs/CC electrode exhibits high performance with a low onset overpotential of 33 mV, a Tafel slope of 70 mV dec<sup>-1</sup> and a Faradaic efficiency of nearly 100%. This catalyst maintain its catalytic activity for at least 30 h and only needs overpotentials of 48 and 190 mV to attain current densities of 10 and 100 mA cm<sup>-2</sup>, respectively.

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## Introduction

Hydrogen is considered as an ideal clean and renewable energy carrier alternative to fossil fuels [1,2]. Electrolysis of water is a simple method to produce high-purity hydrogen

and two half reactions are involved under acidic conditions:  $4\text{H}^+ + 4\text{e}^- \rightarrow 2\text{H}_2$  (cathode reduction) and  $2\text{H}_2\text{O} \rightarrow \text{O}_2 + 4\text{H}^+ + 4\text{e}^-$  (anode oxidation). However, to make the hydrogen evolution reaction (HER) more energy-efficient, an advanced electrocatalyst is required to drive high current density at low overpotential ( $\eta$ ) [3]. Pt-group

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metals are the most active catalysts but suffer from high cost. Proton exchange membrane technology-based water electrolysis units require using catalysts efficiently under strongly acidic conditions [4]. Although abundant nickel based materials are active for hydrogen evolution [5], they suffer from poor corrosion stability in acidic media. As such, it is highly attractive to develop acid-stable non-noble-metal alternatives made of earth-abundance elements.

Cobalt is emerging as an interesting non-noble metal for its catalytic power toward electrochemical hydrogen evolution.  $\text{CoSe}_2$  has been developed as an active HER catalyst in acidic solutions [6,7]. CoP nanoparticles have also proven as an effective acid-stable HER catalyst [8], but the catalyst preparation suffers from the involvement of several organic solvents and multiple tedious steps. We have developed a facile organic solvent-free strategy toward CoP nanocrystals-decorated carbon nanotube (CoP/CNT) as a high-active HER catalyst via low-temperature phosphidation of its  $\text{Co}_3\text{O}_4$ /CNT precursor [9]. All these CoP catalysts are required to be effectively immobilized on current collectors with a polymer binder for electrochemical tests. The polymer binder, however, may block active sites and inhibit diffusion leading to reduced effective catalytic activity [10]. Carbon cloth (CC) woven by carbon fiber exhibits high electronic conductivity and flexibility, offering us a new 3D support to integrate HER catalysts for application in technological devices. Our recent study has shown that direct growth of CoP nanowire arrays on CC (CoP/CC) leads to a high-efficient hydrogen evolution cathode with superior catalytic activity over CoP/CNT [11].

In this communication, we describe our further effort toward this direction in growing CoP nanoparticles film on CC (CoP NPs/CC) through low-temperature phosphidation of its Co NPs/CC precursor. Electrochemical experiments show that the CoP NPs/CC, as a cathode for electrochemical hydrogen evolution in acidic media, exhibits superior catalytic activity and stability over previously reported CoP/CNT [9] and CoP/CC [11].

## Experimental sections

### Synthesis of CoP NPs/CC

A piece of clean CC (1 cm × 1 cm, Wuhan Instrument Surgical Instruments Business) was dipped into a homogenous ink containing 4 g  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ , 0.5 g polyvinylpyrrolidone (PVP), and 5 mL dimethylformamide (DMF), and withdraw from the ink and dried at 80 °C for 0.5 h. The resulting product was calcined at 600 °C for 2 h in Ar flow, leading to Co NPs/CC. To prepare CoP NPs/CC, 3 pieces of Co NPs/CC and 0.3 g of  $\text{NaH}_2\text{PO}_2$  were put at two separate positions in a porcelain boat with  $\text{NaH}_2\text{PO}_2$  at the upstream side of the furnace. Subsequently, the samples were heated at 300 °C for 120 min in an Ar atmosphere, and then naturally cooled to ambient temperature. The CoP loading was determined to be 4.0 mg  $\text{cm}^{-2}$  with the use of a high precision microbalance. CoP NPs/CC with different CoP loading of 2.4 and 6.3 mg  $\text{cm}^{-2}$  were similarly obtained by changing the concentration of Co precursor in the ink.

## Characterizations

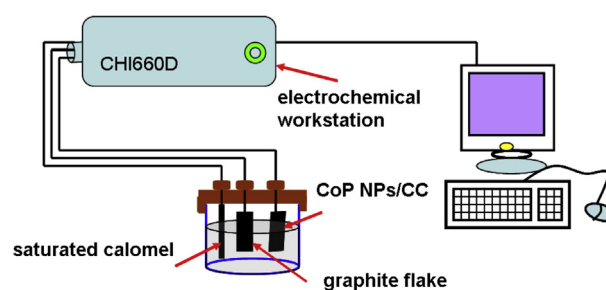
Powder X-ray diffraction (XRD) data were recorded on a RigakuD/MAX 2550 diffractometer. Scanning electron microscope (SEM) measurements were made on an XL30 ESEM FEG microscope. Transmission electron microscopy (TEM) measurements were made on a Hitachi H-8100 electron microscope (Hitachi, Tokyo, Japan). The generated gas was confirmed by gas chromatography (GC) analysis and measured quantitatively using a calibrated pressure sensor to monitor the pressure change in the cathode compartment of an H-type electrolytic cell [9,11].

## Electrochemical measurements

All electrochemical measurements were carried out with a CHI660D electrochemical analyzer (CH Instruments, Inc., Shanghai) in a three-electrode system using CoP NPs/CC as the working electrode, saturated calomel electrode (SCE) as the reference electrode, and a graphite flake as the counter electrode (Fig. 1). Linear sweep voltammetry measurements (LSVs) were conducted in 0.5 M  $\text{H}_2\text{SO}_4$  at a scan rate of 2 mV  $\text{s}^{-1}$ . Cyclic voltammeteries (CVs) were conducted at 100 mV  $\text{s}^{-1}$ . All the electrochemical data are presented without iR compensation. All the potentials reported in our work are versus the reversible hydrogen electrode (RHE). In 0.5 M  $\text{H}_2\text{SO}_4$ ,  $E(\text{RHE}) = E(\text{SCE}) + (0.242 + 0.059 \text{ pH})$ .

## Results and discussion

Fig. 2a shows the XRD patterns of Co NPs/CC (blue curve) and CoP NPs/CC (red curve). The strong diffraction peak at 27° arises from the CC substrate. The diffraction peaks of the precursor can be well ascribed to the phase of metallic Co (JCPDS No. 15-0806) and the phosphided product only shows diffraction peaks corresponding to orthorhombic CoP (JCPDS No. 65-2593), indicating the successful conversion of metallic Co to CoP via low-temperature phosphidation reaction. The SEM images of Co NPs/CC (Fig. 2b) shows that the entire surface of the CC was covered with Co NPs. The subsequent phosphidation leads to CoP NPs film coated CC (Fig. 2c). The corresponding energy dispersive X-ray (EDX) spectrum (Fig. 2d) reveals the atomic ratio between Co and P is close to 1:1. These observed C and O peaks originate from CC and oxidized species of CoP [9,11,12], respectively. The high-



**Fig. 1** – Schematic diagram illustrating the CHI660D electrochemical analyzer and operating conditions.

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