Supporting Information Available

Graphene/porous cobalt nanocomposite and its noticeable electrochemical hydrogen storage ability at room temperature

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Experimental section

XRD analyses were performed on a X’Pert Pro diffractometer with Cu Kα radiation (λ =1.54Å). The surface morphology of the samples was observed on a scanning electron microscopy (JEOL-JSM-6700F) and a transmission electron microscopy (JEOL 2010). BET surface area and pore volume were tested with a Quantachrome Instruments NOVA4000 after the samples were vacuum dried at 300 °C over 10 h. Raman spectra were collected with a JY-HR800 Raman spectrometer (JY Co., France) with an excitation beam wavelength at 532 nm. X-ray photoelectron spectroscopy (XPS) measurements were carried out using a spectrometer with Mg Kα radiation (ESCALAB 250, Thermofisher Co.). The binding energy was calibrated with the C 1s position of contaminant carbon in the vacuum chamber of the XPS instrument (284.8 eV).
**Figure S1** TEM image of graphene used in this work

**Figure S2** Structural characterization of the graphene/Co$_3$O$_4$ composite. (a) XRD pattern, (b) SEM image, (c) TEM image, and (d) HRTEM image, the inset: SAED pattern.
Figure S3 (a) Nitrogen adsorption and desorption isotherms of graphene powder and (b) the corresponding pore-size distribution calculated by BJH method from the desorption branch of graphene powder.

Figure S3 shows the nitrogen adsorption-desorption isotherm and the corresponding Barret-Joyner-Halenda (BJH) pore size distribution curve of the graphene powder. The measured Brunauer-Emmett-Teller (BET) area of the graphene powder is 516.7 m² g⁻¹. The average pore diameters of graphene/cobalt is 1.67 nm, calculated from the desorption branch of the nitrogen isotherm with the BJH method. The corresponding BJH desorption cumulative pore volumes is 0.195 cm³ g⁻¹.
Figure S4 Photograph of the solution obtained without the graphene.

Figure S5 The Co 2p spectra of graphene/cobalt nanocomposite and commercial cobalt powder samples.

Although the XRD pattern (Fig. 1a) shows that Co$_3$O$_4$ in the composite was transformed into metallic cobalt after the thermal treatment at 623 K for 1 h under Ar/H$_2$ gas flow, however, the XPS spectrum (Fig. S5) exhibits that the surface cobalt is largely present as cobalt oxide with the Co 2p$_{3/2}$ at about 780.5 eV. It indicates
that the surface of the graphene/cobalt nanocomposite is easily oxidized due to the small particle size and high surface area, which is similar to that of commercial cobalt powder.

![Graphene/Cobalt Raman Spectra](image)

**Figure S6** Raman spectra of the graphene/cobalt nanocomposite, graphene and commercial cobalt powder samples.

The Raman spectra of the graphene/cobalt nanocomposite show two broad bands at about 1349 and 1589 cm\(^{-1}\), respectively. The band at 1589 cm\(^{-1}\) can be assigned to the \(E_{2g}\) mode of graphite, while the band centered at 1349 cm\(^{-1}\) is the disorder-induced peak characteristics of highly defective graphite.\(^{S2,S3}\)

**References:**

