One-step hydrothermal preparation of bilayer films of NiCo LDH/Pt loaded on Nickel foam surface for HER catalyst activity

*Qingyi Qian, [a,b] Guomin Yu,[c] Qiuping Zhao,[*a] Xingkai Zhang,[*b]*

[a] College of Petrochemical Technology, Lanzhou University of Technology, Lanzhou 730050, China.

[b] State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, China.

[c] Chemical Engineering College, Qinghai University, Xining, 810016, China.

E-mail: zqp_lz@163.com; xingkaizhang@licp.cas.cn

1.Material preparation

1.1 Synthesis of NiCo LDH

Ni foam with a size of 30 mm×50 mm was cleaned by 3 M HCl aqueous solution for 10 min to remove surface oxide layer. The ethanol and deionized water were used to wash the foam to ensure surface cleaning. Next, 0.2 g $CoCl_2$·6 H_2O, 0.1 g $NiCl_2$·6H_2O, 50 mg urea and 400 µL HNO_3 (0.5 M) were dissolved in 50 ml deionized water. The solution was mixed for 30 min, and then the Ni foam was immersed in the solution, followed by stirring for 30 min. The solution and the Ni foam were then transferred to a 50 mL Teflon-lined stainless-steel autoclave and sealed to keep at 180 °C for 12 h, and then it was naturally cooled to room temperature. The obtained sample was washed and dried.

1.2 Synthesis of NiCo LDH /Pt

NiCo LDH/Pt was obtained by the same process except adding platinum salt in the above solution. In order to optimize the Pt doping, different amounts of K_2PtCl_6 (10 g l^{-1}) (250, 450, 550 µL) were evenly dispersed in solutions, the solution was mixed 30 min, and then the Ni foam was immersed in the solution and stirred for 30 min. The semi-finished sample before the hydrothermal reaction was recorded as semi-finished- platinum (SF-Pt) samples. Finally, NiCo LDH /Pt was prepared on SF-Pt by hydrothermal method.

2. Materials Characterization

2.1 Characterization of NiCo LDH, SF-Pt and NiCo LDH/Pt

The surface morphology, composition, structure of the as-prepared NiCo LDH and NiCo LDH /Pt were characterized by the field emission scanning electron microscopy equipped with energy dispersive X-ray detector (FESEM, Apreo S, Thermo Scientific). The crystalline phase of the samples was analysed by high-resolution transmission electron microscopy (HRTEM, Tecnai F30, FEI), energy dispersive X-ray detector (Apreo S, Thermo Scientific). The chemical composition and state of these samples were investigated by the X-ray photoelectron spectroscopy (XPS, ESCALAB- 250Xi, Thermo Scientific). The binding energy of the XPS results was calibrated with C 1s peak (284.8 eV) of surface adventitious carbon as the reference. respectively.

2.2 Electrochemical properties

All electrochemical measurements were carried out in a three-electrode system on μAutolab III, Metrohm workstation, the sample was utilized as the working electrodes. Ag/AgCl (saturated KCl) was used as the reference electrode. Platinum plate was used as the counter electrodes perform for hydrogen evolution reaction (HER). The HER experiment was performed in 1M KOH aqueous electrolyte with iR compensation. Linear sweep voltammograms (LSV) measurements were conducted from −1.5 V to −0.5 V (vs. Ag/AgCl) with a scan rate of 5 $mV \cdot s^{-1}$ in 1.0 M KOH aqueous at room temperature. Potentials are reported against RHE which in turn was calculated using the equation E(RHE) = E(Ag/AgCl) + 0.197 + 0.0592 × pH. Cyclic voltammograms (CV) cycles from -0.9 V to -0.8 V were used to evaluate the sample stability. The Electrochemical Impedance Spectroscopy (EIS) was measured with frequency range from 100 kHz to 0.1 Hz at -1.3 V vs. Ag/AgCl.

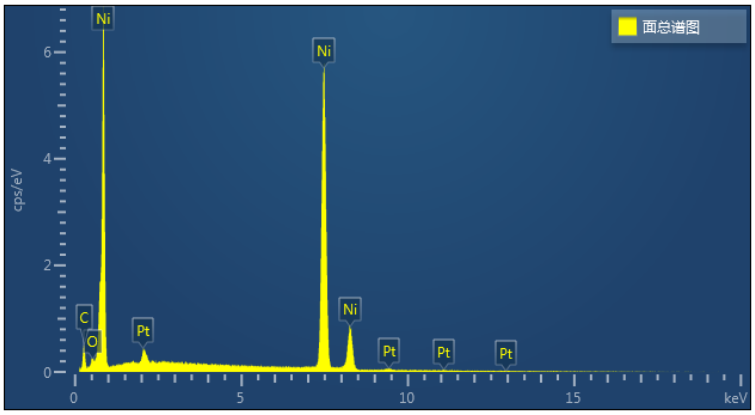

Fig. S1 EDX spectrum of the SF-Pt

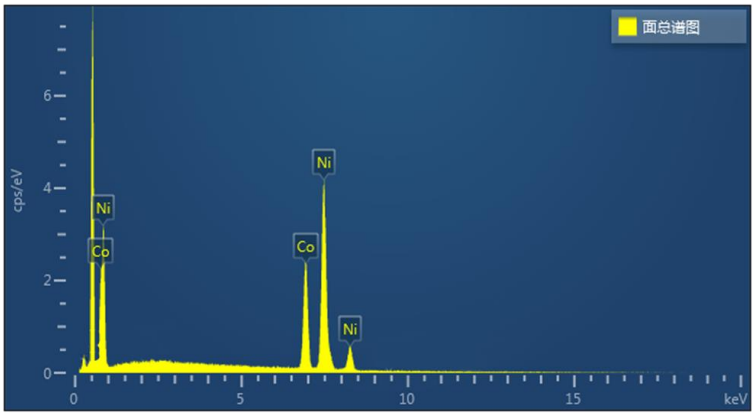

Fig. S2 EDX spectrum of the NiCo LDH/Pt-450