**Electronic Supplementary Information for** 

Boosting supercapacitive performance of ultrathin mesoporous NiCo<sub>2</sub>O<sub>4</sub> nanosheet arrays by surface sulfation



Figure S1 XRD spectrum for the as-deposited sample on Ni foam

The as-deposited sample is denoted as p-NiCo<sub>2</sub>O<sub>4</sub>, p-S0.25M and P-S0.5M, respectively. The four main peaks at  $22.4^{\circ}$ ,  $34.0^{\circ}$ ,  $38.5^{\circ}$ ,  $59.2^{\circ}$  correspond to a rhombohedral structure of Co–Ni layered double hydroxides.<sup>1</sup>

The average crystalline sizes of all samples are calculated by Scherrer Equation<sup>2</sup> and combined with XRD data (1) as following:

$$D_{hkl} = \frac{K\lambda}{B_{hkl}\cos\theta}$$

 $D_{hkl}$  is the crystallite size in nanometers, hkl are the Miller indices of the lattice planes being analyzed.  $\lambda$  is the wavelength of X-ray, taken as 0.154056 nm.  $B_{hkl}$  is the peak width of the X-ray diffraction peak profile at half-maximum height in radians and K is the numerical crystallite-shape factor, normally taken as 0.9 and  $\theta$  is the Bragg angle. Full Width Half Maximum (abbreviated as FWHM) is the  $B_{hkl}$  in degrees. In order to acquire relatively accurate crystallite size, the (311) plane with most prominent diffraction peak is choosed for calculation.

Table S1. The crystalline sizes of the samples

Sample Name	2θ (degree)	FWHM (degree)	D <sub>311</sub> (nm)
Pristine NiCo <sub>2</sub> O <sub>4</sub>	36.894	0.655	12.02
S0.25M	36.668	0.723	11.45
S0.5M	36.688	0.719	11.51



Figure S2 High-magnified SEM images of (a) NiCo<sub>2</sub>O<sub>4</sub>, (b) S0.25M and (c) S0.5M after 5000 cycling tests.



Figure S3 TEM images and the inset SAED pattern of (a)  $NiCo_2O_4$ , (b) S0.25M and (c) S0.5M after 5000 cycling tests.



Figure S4 The core-level S 2p spectra of (a) S0.25M and (b) S0.5M at different stages including before cycling tests, after fully activated.

## References

- 1. L. Qian, L. Gu, L. Yang, H. Yuan and D. Xiao, Nanoscale, 2013, 5, 7388-7396.
- 2. U. Holzwarth and N. Gibson, Nature nanotechnology, 2011, 6, 534-534.