Hydrothermal Synthesis of Nanostructured Flower-like $Ni(OH)_2$ Particles and their Excellent Sensing Performance towards Low Concentration HCN Gas

Mingzhen Hu[a,b], Junhui He[a,*], Mingqing Yang[a], Xiaochun Hu[c], Chunxiao Yan[c] and Zhenxing Cheng[c]

[a] *Functional Nanomaterials Laboratory, Center for Micro/Nanomaterials and Technology and Key Laboratory of Photochemical Conversion and Optoelectronic Materials, Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Zhongguancundonglu 29, Haidianqu, Beijing 100190, China*

[b] *University of Chinese Academy of Sciences, Beijing 100049, China*

[c] *The No.3 Department, Institute of Chemical Defence, P.O.Box 1048, Beijing 102205, China*

[*] Corresponding author. Fax: +86 10 82543535. E-mail address: jhhe@mail.ipc.ac.cn.

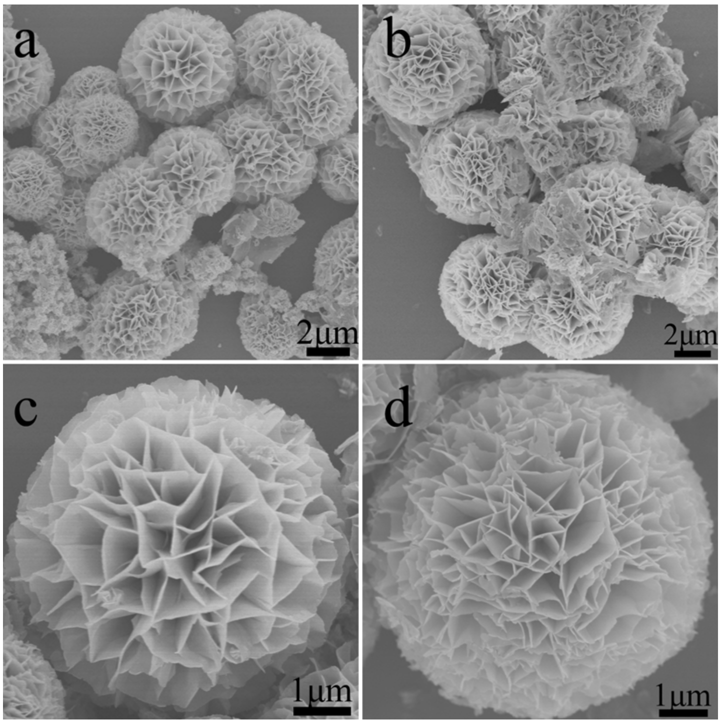


Fig. S1 Typical SEM images of $Ni(OH)_2$ products fabricated at varied hydromal temperatures under otherwise identical conditions (SDBS/Ni molar ratio: 0.1, reaction time: 7 h): (a,c) 80 ℃, (b,d) 140 ℃.

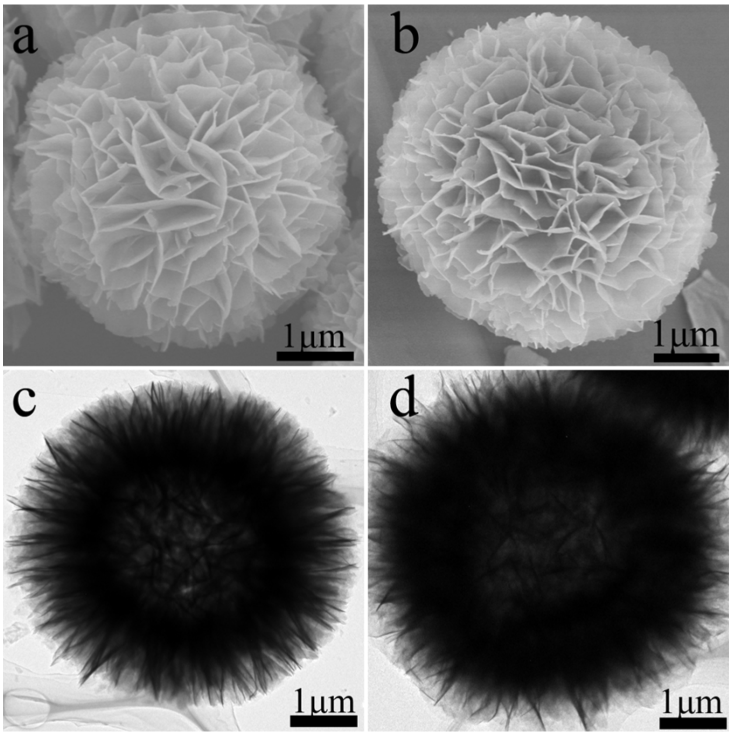


Fig. S2 Typical SEM and TEM images of $Ni(OH)_2$ products synthesized using varied periods of reaction time under otherwise identical conditions (SDBS/Ni molar ratio: 0.1, hydromal temperature: 100 ℃): (a,c): 3 h, (b,d): 12 h.

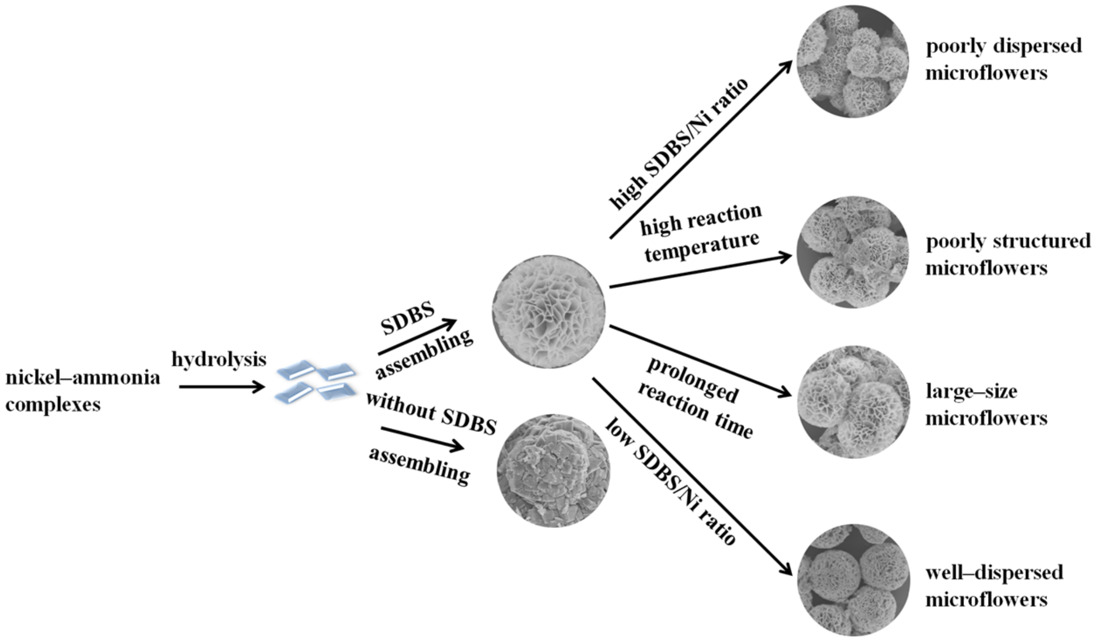


Fig. S3 Schematic illustration of the formation of hierarchically structured $Ni(OH)_2$ particles.

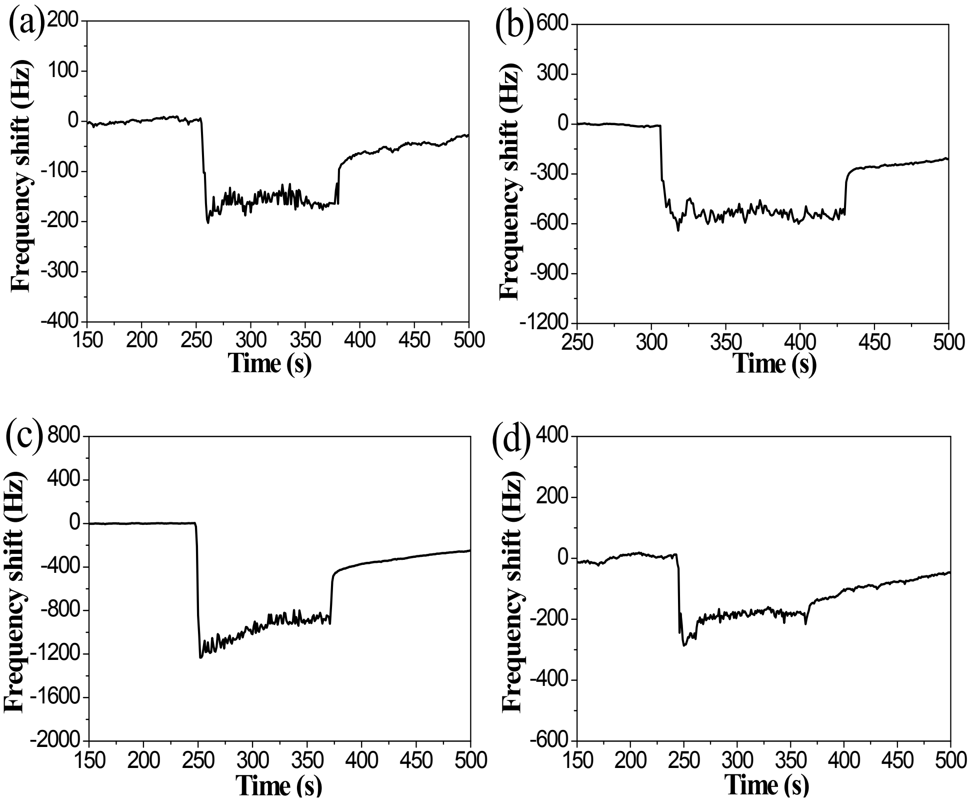
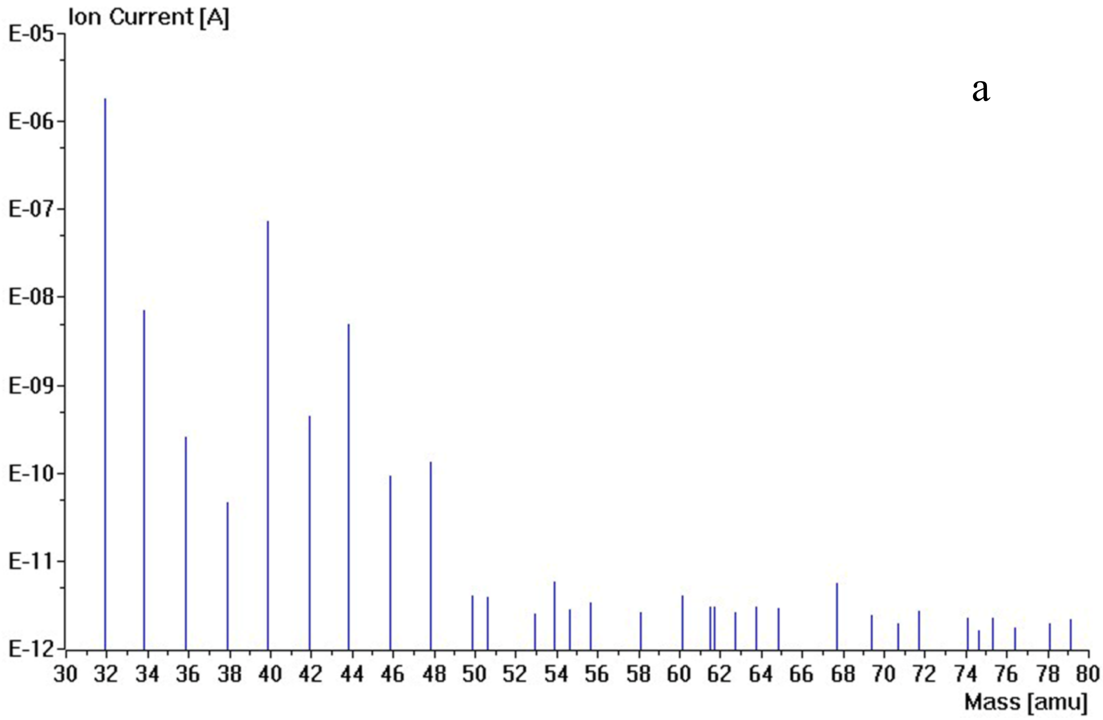


Fig. S4 Response profiles of $Ni(OH)_2$ modified QCM resonator towards: (a) acetone, (b) ethyl ether, (c) water, (d) ethanol. The $Ni(OH)_2$ was prepared using a SDBS/Ni molar ratio of 0.1 at 140 ℃ for 7 h.



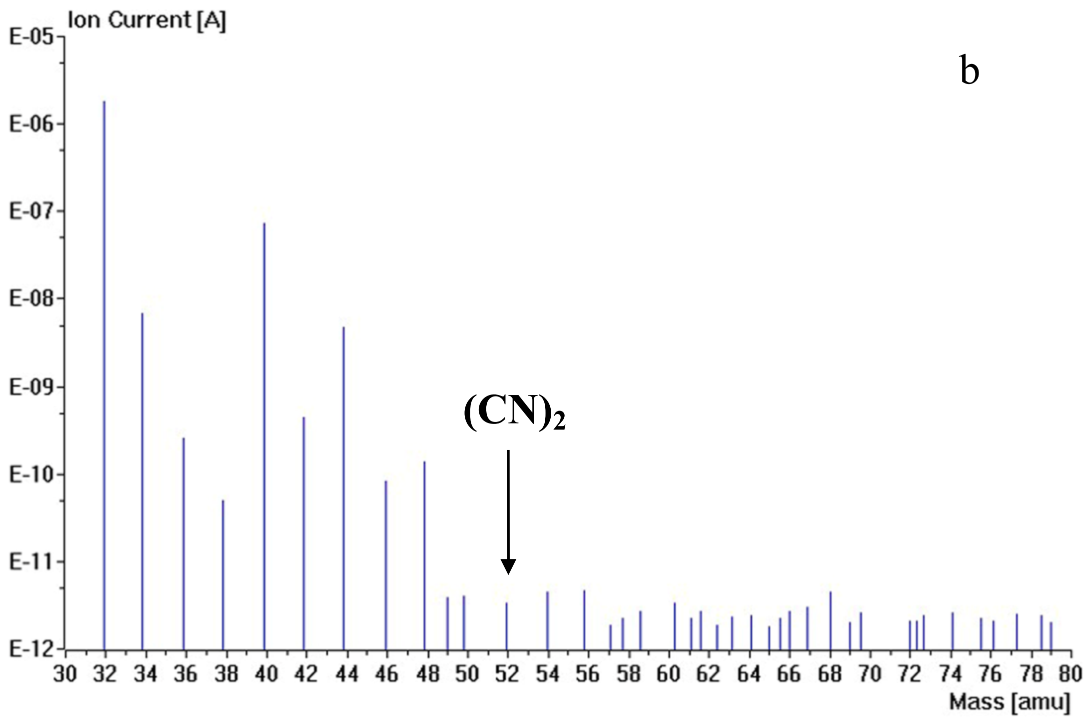


Fig. S5 Mass spectra of HCN effluent before (a) and after (b) contact with $Ni(OH)_2$ modified QCM resonator.

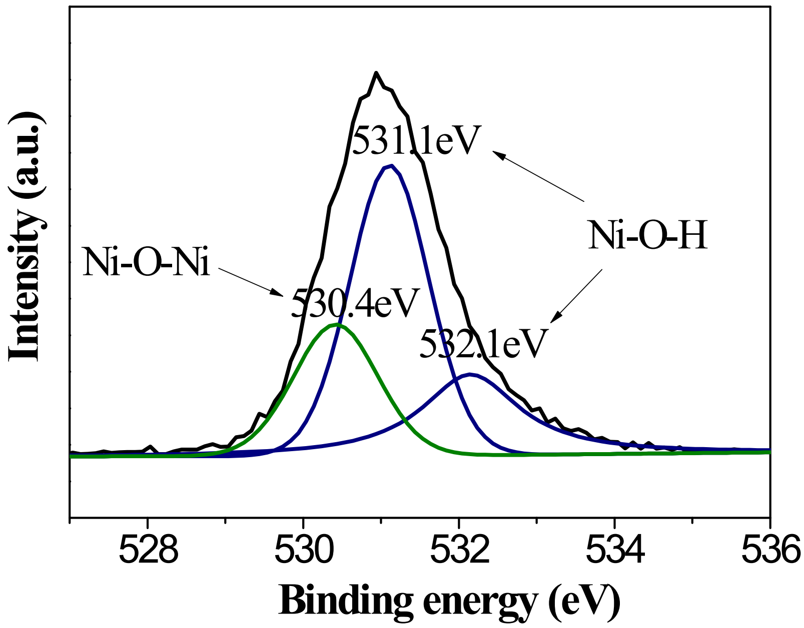


Fig. S6 XPS O 1s spectra of $Ni(OH)_2$ prepared using a SDBS/Ni molar ratio of 0.1. The hydrothermal temperature and reaction time are 100 ℃ and 7 h, respectively.

Calculation of the active oxygen molecules mole number

1. Estimated by calculating the vacancy sites mole number of coated $Ni(OH)_2$ from XPS measurements

The surface molar ratio of Ni/vacancy sites is estimated to be 2:1 from XPS measurements (Fig. 7b). Thus, the active oxygen molecules mole number can be obtained by the following equation:

$$n_{o_2} = \frac{n_{Ni(OH)_2}}{2} = \frac{m_{Ni(OH)_2}}{2 \times M}$$

Where *m (g)* is the coating mass of $Ni(OH)_2$, *M* is the molar mass of $Ni(OH)_2$.

The coating mass of $Ni(OH)_2$ is 6 μg and the molar mass of $Ni(OH)_2$ is 92.7 g/mol. Therefore, the mole number of active oxygen molecules is calculated to be 3.24×10^{-8} mol.

2. Estimated by calculating the vacancy sites mole number of coated $Ni(OH)_2$ from QCM measurements

From the Sauerbrey equation, we can obtain the mass decrease of coated $Ni(OH)_2$. If this mass decrease is due to the removal of active oxygen molecules, we can then obtain the mole number of active oxygen molecules from the followintg equation:

$$n_{o_2} = \frac{A \times \Delta F}{-2.26 \times 10^{-6} \times F_0^{\,2} \times M}$$

Where A *(cm²)* is the sensing surface area, ΔF *(Hz)* is the frequency shift from initial HCN contact to sensing equilibrium, F_0 *(Hz)* is the base frequency of quartz crystal, M is the molar mass of oxygen molecule.

From the above parameters, where A = 0.39 cm^2, ΔF = 1011 Hz, F_0 = 9001274.5 Hz, M = 32 g/mol, we can therefore calculate the mole number of active oxygen molecules as 6.73×10^{-8} mol.