Supporting Information to

# Electrodeposition of nickel nanostructures using silica

# nanochannels as confinement for low-fouling enzyme-free

# glucose detection

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#### S1. Electrochemical characterization of the SNM/FTO electrode

In order to prove the integrity of the SNM/FTO electrode, we examined the electrode characteristics by comparing the electrochemical behavior of three redox probes, namely anionic  $Fe(CN)_6^{3-}$ , cationic  $Ru(NH_3)_6^{3+}$  and nonionic FcMeOH. We compared CVs of the bare FTO electrode, SNM/FTO electrode before and after removal of surfactant micelles from nanochannels (designated as SMs@SNM/FTO and SNM/FTO for discrimination, respectively). The results are shown in Fig. S1. Faradic current with apparent voltammetric peaks was only observed with nonionic FcMeOH at the SMs@SNM/FTO electrode, indicating the FTO surface was fully covered by the SNM without cracking or leakage and that CTAB micelles filled all nanochannels to block the mass transport of ionic redox probes. In contrast, after removal of CTAB micelles, redox current was observed for all probes. Furthermore, the current magnitude of Ru(NH<sub>3</sub>)<sub>6</sub><sup>3+</sup> was apparently larger than that of Fe(CN)<sub>6</sub><sup>3-</sup>. This permselective behavior can be ascribed to the ultrasmall size of silica nanochannels (2 ~ 3 nm in diameter) and their negatively charged surface due to deprotonation of surface silanol groups.



**Fig. S1.** Comparing electrochemical responses of bare FTO, SMs@SNM/FTO and SNM/FTO electrodes in 0.1 M KCl solution containing three different redox probes: (a) 0.5 mM  $K_3$ Fe(CN)<sub>6</sub>, (b) 0.5 mM Ru(NH<sub>3</sub>)<sub>6</sub>Cl<sub>3</sub>, (c) 0.5 mM FcMeOH.

### S2. Raman spectrum of the Ni<sub>40</sub>@SNM/FTO electrode

**Fig. S2** shows the Raman spectra of the Ni<sub>40</sub>@SNM/FTO and SNM/FTO recorded in the range of  $300 \text{ cm}^{-1} - 1500 \text{ cm}^{-1}$ . Two prominent peaks of NiO around 573 cm<sup>-1</sup> and 1102 cm<sup>-1</sup> were observed for the Ni<sub>40</sub>@SNM/FTO, corresponding to one-phonon first-order longitudinal-optical mode and two-phonon secondorder longitudinal-optical mode, respectively.<sup>S1, S2</sup>



Fig. S2. Raman spectra of the Ni<sub>40</sub>@SNM/FTO and SNM/FTO.

# S3. CVs of the Ni<sub>40</sub>@SNM/FTO electrode in 0.1 M PBS (pH = 7) in the

## absence and presence of glucose

**Fig. S3** shows the cyclic voltammograms of the Ni<sub>40</sub>@SNM/FTO electrode in 0.1 M PBS (pH = 7) in the absence and presence of 5 mM glucose. The Ni<sub>40</sub>@SNM/FTO electrode displayed poor current responses to glucose under neutral condition.



**Fig. S3.** Cyclic voltammograms of the Ni<sub>40</sub>@SNM/FTO electrode in 0.1 M PBS (pH = 7) in the absence (dotted curve) and presence (solid curve) of 5 mM glucose.

### S4. Optimization of glucose detection potential

We conducted electrochemical detection of glucose with the Ni<sub>40</sub>@SNM/FTO electrode. **Fig. S4** showed the chronoamperometric responses of the electrode to the successive addition of glucose at four different potentials. The current magnitude displayed at 0.65 V is apparently larger than that at others, but the positive offset of baseline is also very big. So eventually 0.6 V was considered to be the optimal potential for glucose detection.



**Fig. S4.** Chronoamperometric responses of the Ni<sub>40</sub>@SNM/FTO electrode to the successive addition of 0.5 mM glucose in 0.1 M NaOH at three different potentials.

## S5. SEM characterization of the Ni@FTO electrode

The Ni/FTO electrode was prepared by directly electrodeposition of nickel on a bare FTO electrode surface. The electrode morphology was characterized by SEM. As shown in **Fig. S5**, the SEM image of Ni@FTO electrode with an electrodeposition time of 40 s revealed three layers from top to down, i.e., nickel layer, FTO layer and glass.



Fig. S5. SEM image showing the cross-section of Ni/FTO electrode.

#### S6. The selectivity of the Ni<sub>40</sub>@SNM/FTO electrode

We selected six interferents with different molecular weights to study the selectivity of Ni<sub>40</sub>@SNM/FTO electrode. The chronoamperometric responses of the Ni<sub>40</sub>@SNM/FTO electrode upon successive addition of glucose (0.25 mM) and mixture (0.25 mM glucose and 0.25 mM interference FA/AP/UA/Ser/Gly, 0.25 mM glucose and 2.5  $\mu$ M AA) to 0.1 M NaOH at +0.6 V were recorded. (**Fig. S6 a-f**).



**Fig. S6.** (a-f) Chronoamperometric responses of the Ni<sub>40</sub>@SNM/FTO electrode upon successive addition of glucose (0.25 mM) and mixture (0.25 mM glucose and 0.25 mM interferent FA/AP/UA/Ser/Gly, 0.25 mM glucose and 2.5  $\mu$ M AA) to 0.1 M NaOH at +0.6 V.

### S7. Detection of glucose in blood samples

The practical application of the Ni<sub>40</sub>@SNM/FTO electrode was tested by determining the glucose concentration in the human blood sample. In this determination process, 250  $\mu$ L of blood sample was added into 25 mL of 0.1 M NaOH solution and the chronoamperometric response was 8.76  $\mu$ A.



Fig. S7. Chronoamperometric response of the Ni<sub>40</sub>@SNM/FTO electrode upon the addition of blood sample (250  $\mu$ L) in 0.1 M NaOH (25 mL) at +0.6 V.

## References

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