A facile electrochemical approach to fabricate a nanoporous gold film electrode and its electrocatalytic activity towards dissolved oxygen reduction

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Electronic Supplementary Information (ESI)

Experimental section

Reagents

Sulfuric acid and potassium nitrate (Merck) were used as received and Milli-Q water was used in all experiments. 0.5 mol L\(^{-1}\) H\(_2\)SO\(_4\) and 0.1 mol L\(^{-1}\) KNO\(_3\) solutions were used as supporting electrolyte for the fabrication of NPGF electrode and in studies on the electrocatalytic dissolved oxygen reduction, respectively.

Apparatus

The electrochemical measurements were performed using a PalmSens portable electrochemical analyzer (BV, Netherland) employing a conventional three electrode cell system: the electrochemically fabricated NPGF onto a gold electrode (geometrical area 0.07065 cm\(^2\)), a platinum foil and Ag/AgCl (saturated KCl) as working, counter and reference electrodes, respectively. All experiments were performed at ambient temperature (298K). FE-SEM images of the NPGF electrode were taken using a JOEL JSM740F equipment with acceleration voltage of 30 kV.

Electrochemical surface area and Roughness factor calculations

The roughness factor (Rf) was calculated using the following formula

\[
Rf = \frac{\text{Electrochemical surface area (ECSA)}}{\text{Geometrical area}}
\]
The electrochemical surface area of NPGF and bare gold electrodes was calculated using the charge associated with the reduction of gold oxide by integration, which is proportional to the real active surface area of the gold surface. (*Pure & Appl. Chem.*, 1991, 63, 711-734; *Chem. Mater.* **2007**, 19, 3648-3653; J Mater Chem, 2012, 22, 6733).

- **ECSA for bare gold electrode**
  
  Charge (Q) = Area / Scan rate  
  = 0.0129 / 0.1  
  = 0.129 mC, which is equal to 129 μC  
  
  ECSA = Q / 390 μC cm\(^{-2}\)  
  = 129 μC / 390 μC cm\(^{-2}\)  
  = 0.3308 cm\(^2\)  
  
  Note: A value of 390 μC cm\(^{-2}\) has been suggested for polycrystalline gold. Ref: *Pure & Appl. Chem.*, 1991, 63, 711-734)

- **ECSA for NPGF electrode (potential held at 2.0 V for 60 min)**
  
  Charge (Q) = Area / Scan rate  
  = 0.2378 / 0.1  
  = 2.378 mC which is equal to 2378 μC  
  
  ECSA = Q / 390 μC cm\(^{-2}\)  
  = 2378 μC / 390 μC cm\(^{-2}\)  
  = 6.097 cm\(^2\)  
  
  Note: In a similar way, ECSA values for NPGF electrodes fabricated in experiments where the potential was held at 10, 20 and 30 min were found to be 1.038, 1.513 and 3.10 cm\(^2\), respectively.

- **Roughness factor (Rf)**
  
  For bare gold electrode  
  Rf = 0.3308 / 0.07065 = 4.68  
  (Geometrical area of the gold electrode = 0.07065 cm\(^2\))
For NPGF electrode

\[ R_f = \frac{6.097}{0.07065} \]

\[ = 86.30 \quad \text{i.e.} \sim 86 \]

Note: In a similar way, roughness factor values for NPGF electrodes fabricated in experiments where the potential was held at 10, 20 and 30 min were found to be 14.5, 21.5 and 44, respectively.

**Fig. S1:** Cyclic voltammogram recorded in a 0.1 mol L\(^{-1}\) KNO\(_3\) solution containing oxygen (saturated) using the NPGF electrode at the scan rate of 0.1 V s\(^{-1}\)