Supporting information

Deciphering the Atomic Scale Electrocatalytic Sites in Hierarchically Porous Gold Nanostructures: Implications for Ascorbic Acid Electrooxidation

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Calculation of electrochemical surface area

At first, mathematical area is calculated by the integration of the reduction peak of the CV of NPG electrode in 0.5 M H$_2$SO$_4$ as shown in Fig. R5.

![Mathematical area estimated by the integration of reduction peak of the CV of NPG prepared at -4 V for 100 s in 0.5 M H$_2$SO$_4$ at scan rate 50 mV s$^{-1}$ on 1.6 mm Au electrode.](image)

Charge consumed in the reduction of gold oxide into gold is calculated by the formula;

Charge (Q) = Peak area / Scan rate

\[
= \frac{82.85 \times 10^{-6} \text{ (A.V)}}{0.05 \text{ (V.s$^{-1}$)}}
\]

\[
= 1667 \times 10^{-6} \text{ A.s (equal to 1667 } \mu\text{C)}
\]

ESA = Q / 390μC cm$^{-2}$

\[
= 1667 \mu\text{C} / 390\mu\text{C cm}^{-2}
\]

\[
= 4.25 \text{ cm}^2
\]

Note: A value of 390 μC cm$^{-2}$ was used for one monolayer charge of gold oxide reduction. Ref: Pure & Appl. Chem., 1991, 63, 711).
Figure S2: XRD pattern of NPG film coated on Au substrate.

Figure S3: SEM image of NPG film recorded at low magnification.
Figure S4: High resolution XPS spectrum of Au4f and its fitting.
Figure S5. SEM image of a 30 nm slice of NPG sample recorded in FSD mode (a). IPF crystal orientation map recorded within the selected region of the sample with reference to viewing direction along y axis (b). The color key of the IPF of gold is indicated on the right side for gold. Critical misorientation angle is 10°.
Figure S6: strain contour recorded within the selected region of the sample.