Supporting information for

## 3D PtAu Nanoframe Superstructure as High-Performance Carbon-

## **Free Electrocatalyst**

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Figure S1 SEM image of TOh PtAu NFs with different thickness. Thickness can be controlled by controlling the amount of  $H_2$ PtCl<sub>6</sub> precursor: (A) 11  $\mu$ M, (B) 22  $\mu$ M, (C) 44  $\mu$ M



Figure S2 UV-Vis spectra of TOh Au NPs (red trace), TOh Au@Pt NPs (black trace), TOh PtAu NFs (blue

trace).



Figure S3 SEM image of self-assembled ordered TOh Au NPs with uniform shape and size.

Increasing the amount of etchant/Decreasing Au Atomic Ratio



**Figure S4** Controlling atomic ratio of PtAu NFs by changing etchant concentration. Bottom figure of each column shows low magnification and top figure shows high magnification of PtAu NFs.

Ele	ctron Image	2 _							
		Costev				Map Sum Spectrum Pt Pt Pt Pt Au Au 10 15 keV			
* 250nm *									
Element	Line Type	Apparent Co ncentration	k Ratio	Wt%	Wt% Sigma	Atomic %	Standard Label	Factory St andard	Standard Cal ibration Date
Element	Line Type L series	Apparent Co ncentration 0.00	k Ratio 0.00003	Wt% 0.39	Wt% Sigma 3.53	Atomic % 0.70	Standard Label	Factory St andard Yes	Standard Cal ibration Date
Element Ag Pt	Line Type L series M series	Apparent Co ncentration 0.00 0.77	k Ratio 0.00003 0.00771	Wt% 0.39 65.55	Wt% Sigma 3.53 3.07	Atomic % 0.70 65.55	Standard Label Ag Pt	Factory St andard Yes Yes	Standard Cal ibration Date
Element Ag Pt Au	Line Type L series M series M series	Apparent Co ncentration 0.00 0.77 0.36	k Ratio 0.00003 0.00771 0.00363	0.39 65.55 34.07	Wt% Sigma 3.53 3.07 2.35	Atomic % 0.70 65.55 33.75	Standard Label Ag Pt Au	Factory St andard Yes Yes Yes	Standard Cal ibration Date

Figure S5 EDS mapping for compositional analysis of a typical TOh PtAu NFs



Figure S6 ECSA of Pt/C, TOh Au@Pt solid NPs and TOh PtAu NFs.



Figure S7 TEM image of PtAu NFs after 800 cycles in  $0.1 \text{ M H}_2\text{SO}_4$ 



**Figure S8** Catalytic performance of TOh NFs (A,D), TOh Au@Pt NPs (B,E) and Pt/C (C,F) with different film thicknesses as-shown in Figure S8. Cyclic voltammogram was carried out using in 0.1 M  $H_2SO_4$  (A,B and C), MOR using 1 M Methanol in 0.1 M  $H_2SO_4$  (D,E and F), respectively.



**Figure S9** SEM images of TOh PtAu NFs (A-C), TOh Au@Pt NPs (D-F), and Pt/C (G-I) with different thicknesses.