

## 1. Supplementary information

### Photo-catalytic hydrogen production over Au/g-C<sub>3</sub>N<sub>4</sub>. Effect of gold particles dispersion, and morphology.

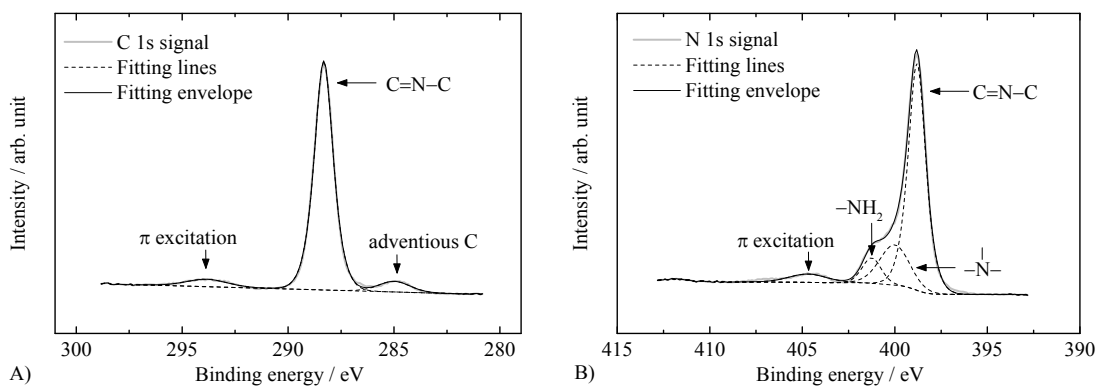
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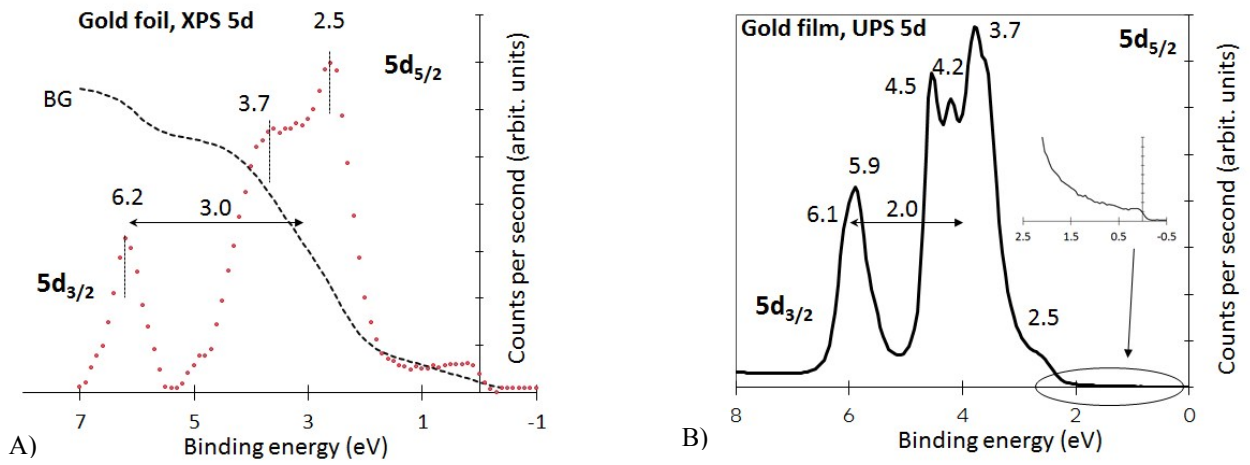
**Figure SI 1: XP spectra of 2.1 wt% Au/g-C<sub>3</sub>N<sub>4</sub> carbon and nitrogen 1s regions**

XP spectra of 2.1 wt% Au/g-C<sub>3</sub>N<sub>4</sub>: A) C 1s and B) N 1s regions.

**Table SI 1: Impact of thermal treatment on gold mean particle size**

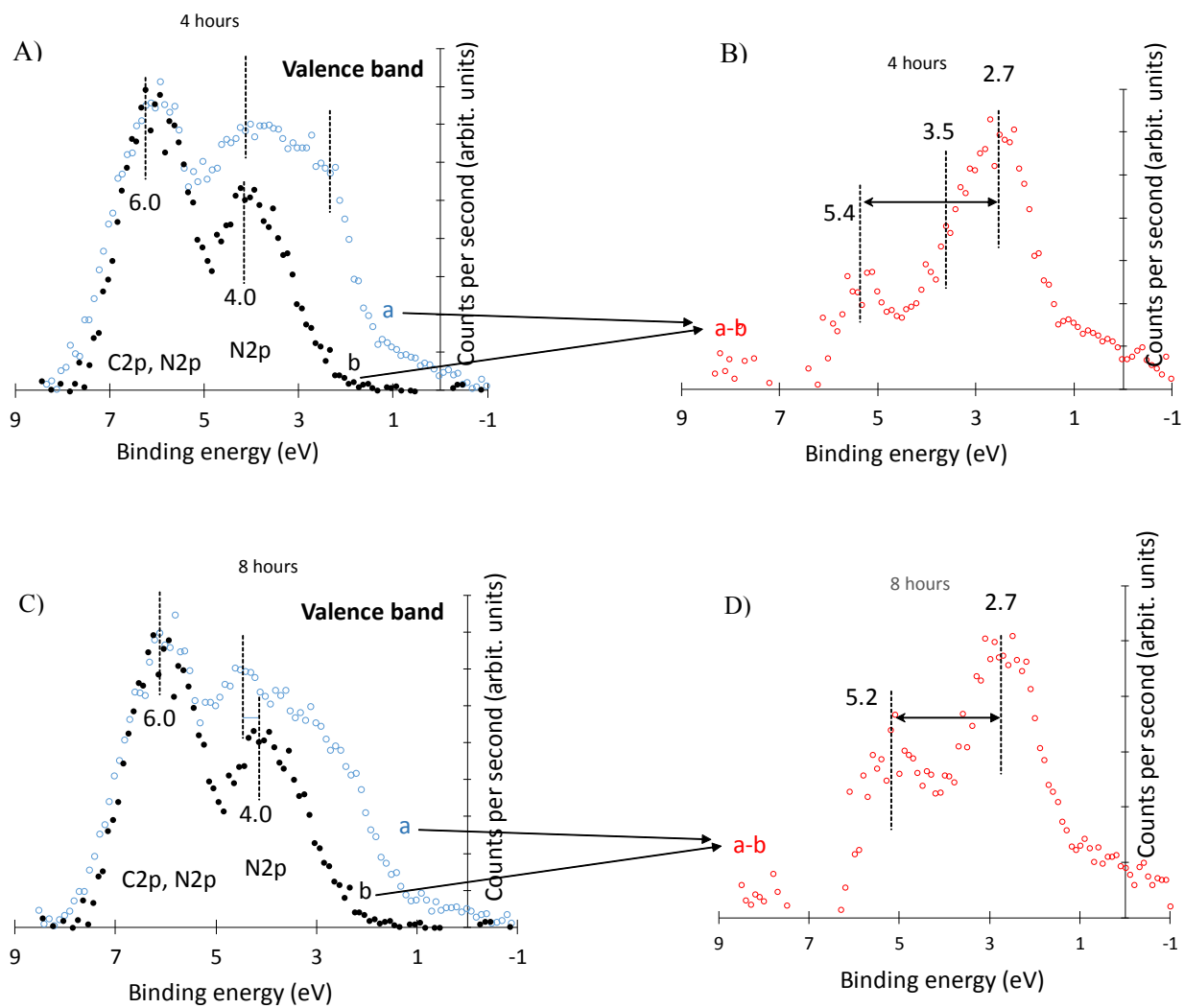
Mean particle sizes extracted from TEM micrographs of 2.1 wt% Au/g-C<sub>3</sub>N<sub>4</sub> (4-hours) before and after thermal treatment in different atmospheres.

Thermal treatment	Mean particle size / nm	Standard deviation / nm
None	1.8	0.6
Static air	5.8	2.9
5 vol% H <sub>2</sub> in argon	4.8	2.0



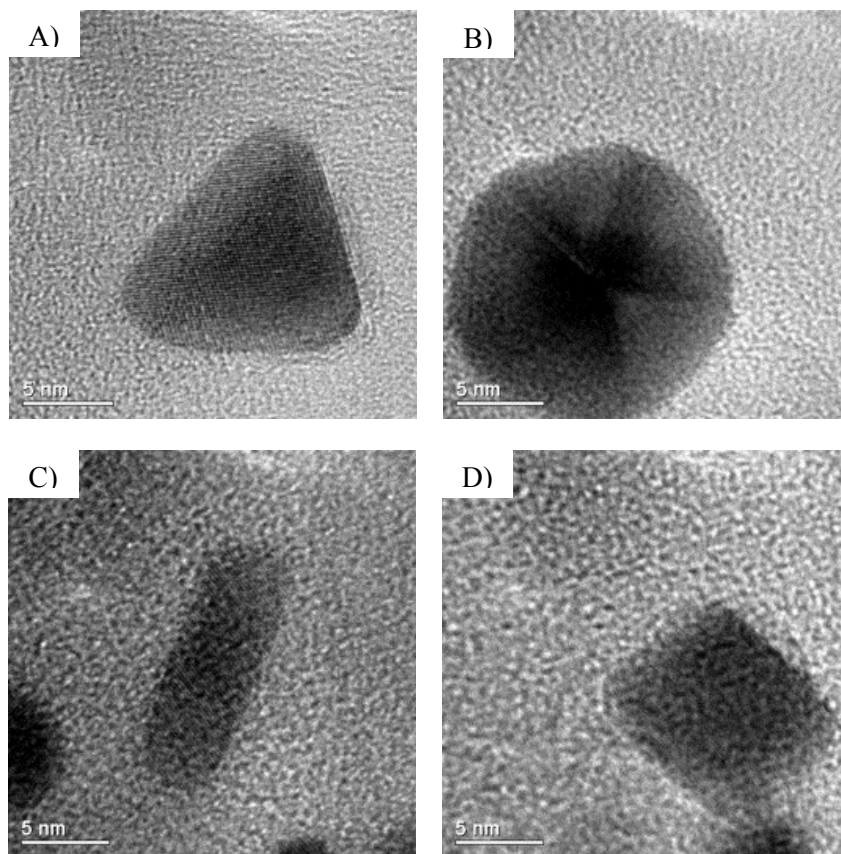
**Figure SI 2: XPS and UPS measurements of Au foil**

A) Valence band XPS of Au foil; B) Valence band UPS of clean (Ar ions sputtered) Au thin film. BG: Shirley background.



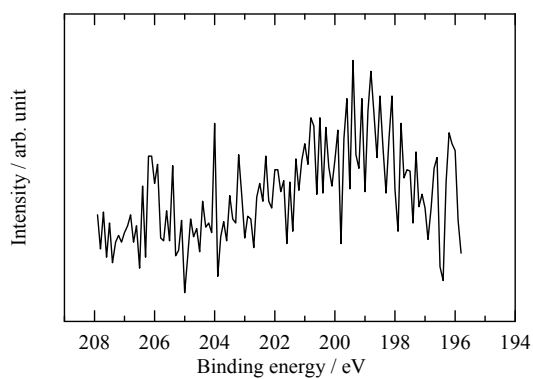
**Figure SI 3: Valence band region of "nominally" 2.1 wt% Au/g-C<sub>3</sub>N<sub>4</sub> catalysts.**

A) Au/g-C<sub>3</sub>N<sub>4</sub> (4-hour deposition time) and g-C<sub>3</sub>N<sub>4</sub>, B) subtracted spectra of A) upon normalization at 6 eV, C) Au/g-C<sub>3</sub>N<sub>4</sub> (8-hour deposition time) and g-C<sub>3</sub>N<sub>4</sub>, D) subtracted spectra of C) upon normalization at 6 eV.



**Figure SI 4: TEM micrographs of 2.1 wt% Au/g-C<sub>3</sub>N<sub>4</sub> after thermal treatment in 5 vol% H<sub>2</sub> in argon**

TEM micrographs of reduced 2.1 wt% Au/g-C<sub>3</sub>N<sub>4</sub> presenting various morphologies such as A) triangular nanoplate, B) polyhedron, C) rod and D) cube.



**Figure SI 5: XPS spectra of chlorine 2p region for as-prepared 2.1 wt% Au/g-C<sub>3</sub>N<sub>4</sub>**

**Table SI 2: Impact of urea concentration on gold mean particle size and gold coverage**

Mean particle sizes extracted from TEM micrographs of 2.1 wt% Au/g-C<sub>3</sub>N<sub>4</sub> (4-hours) prepared with various urea concentrations and reduced in 5 vol% H<sub>2</sub> in argon previous to characterisation. Atomic percentage of gold extracted from XPS measurements.

Urea concentration / M	[Urea]:[Au]	Mean particle size / nm	Standard deviation / nm	Au at% XPS Au4f	Particle density 100nm <sup>2</sup>	<sup>197</sup> Au area % TEM
0	0	3.5	1.7	0.3	0.7	11
0.25	230	5.4	1.9	2.6	2.4	58
0.42	400	4.8	2.0	0.6	1.0	16
0.85	830	5.8	3.1	1.3	1.2	31

a: (number of Au particles x area of the mean particle size / total area) x 100, from TEM images.