

**Pulsed laser synthesis of free-standing Pt single atoms in iced block for enhancing photocatalytic hydrogen evolution of g-C<sub>3</sub>N<sub>4</sub>**

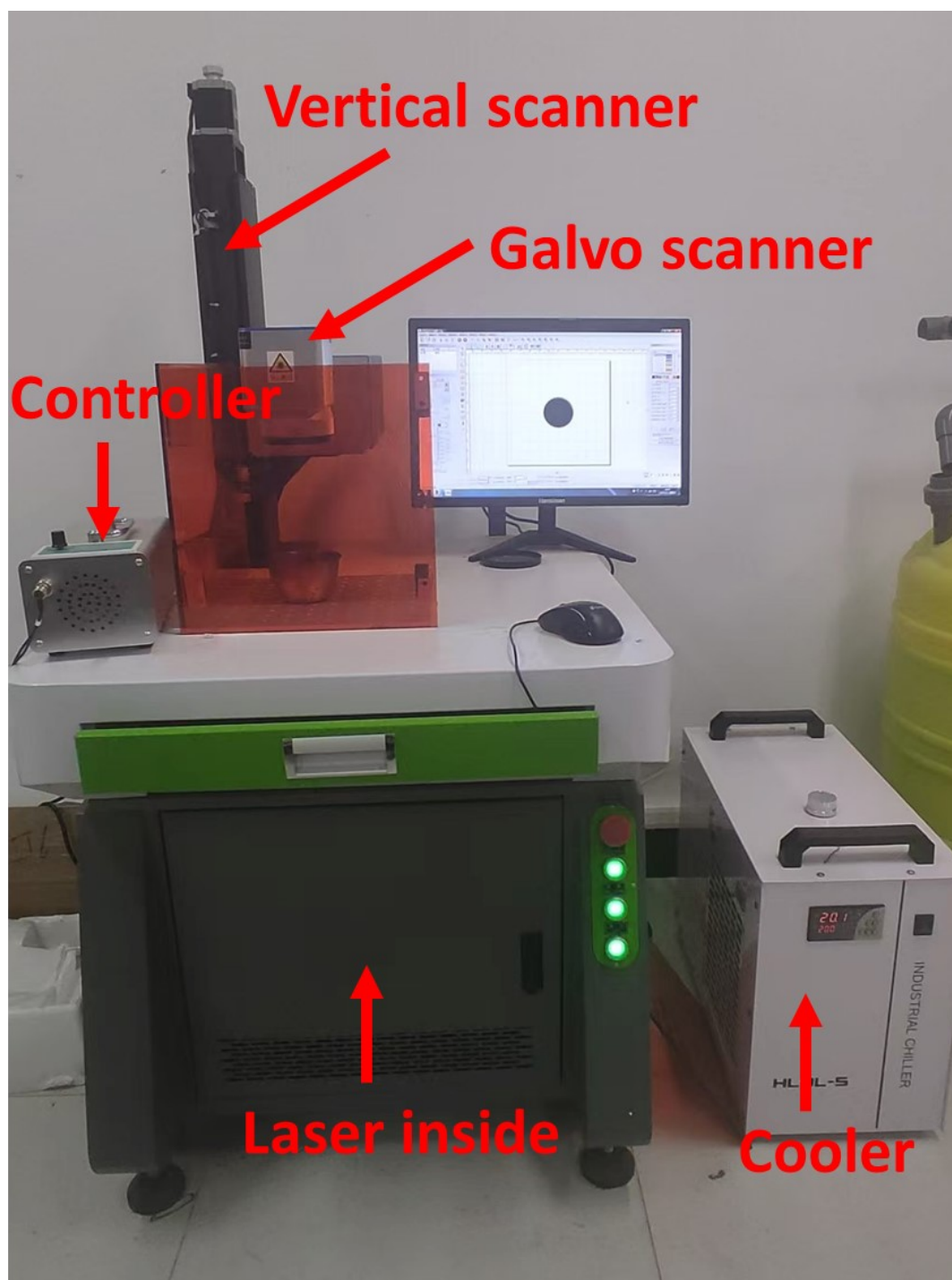
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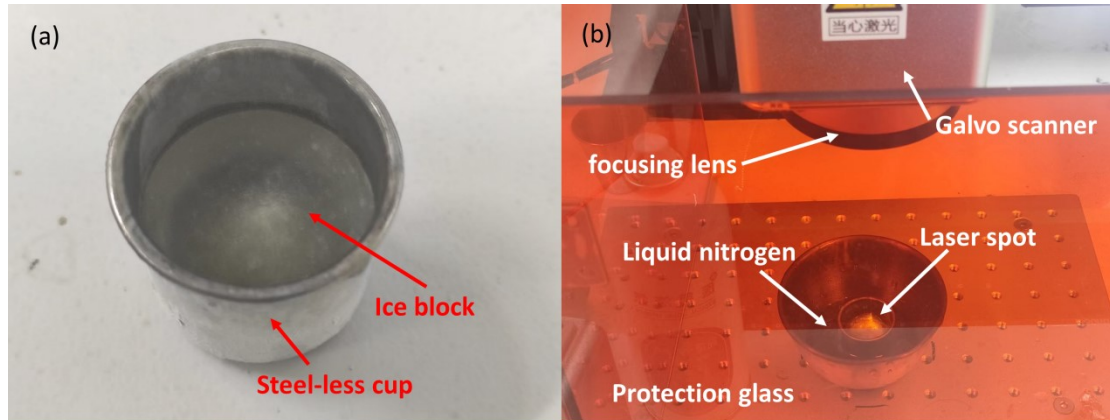
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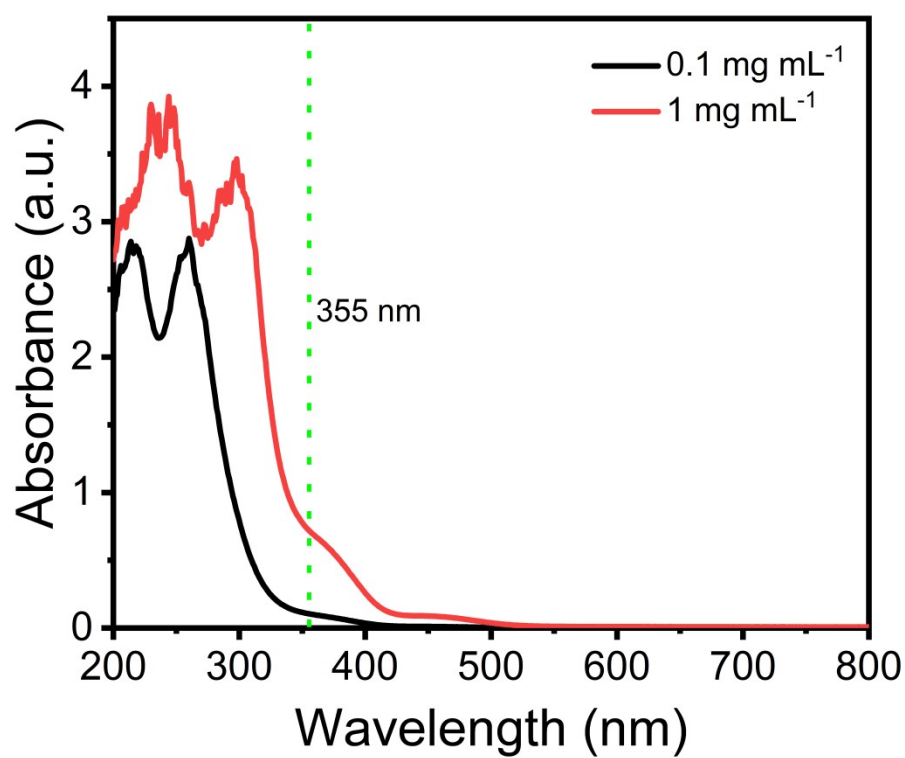
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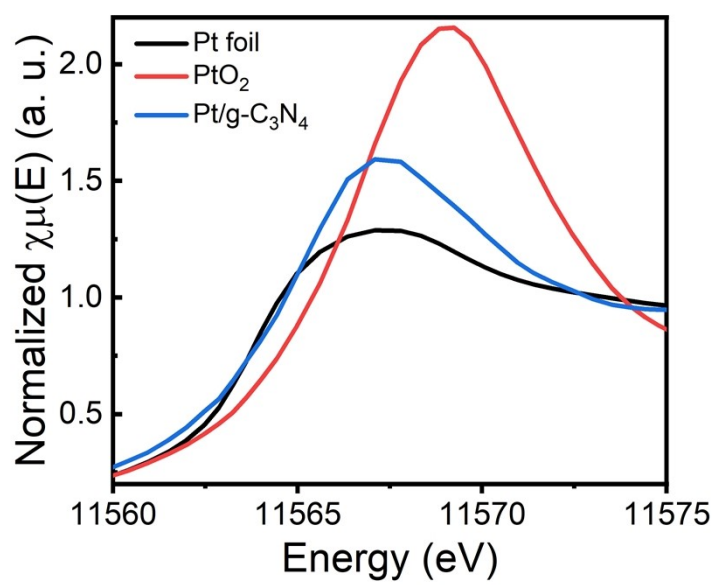
**Fig. S1** The photograph of the laser scanning set.



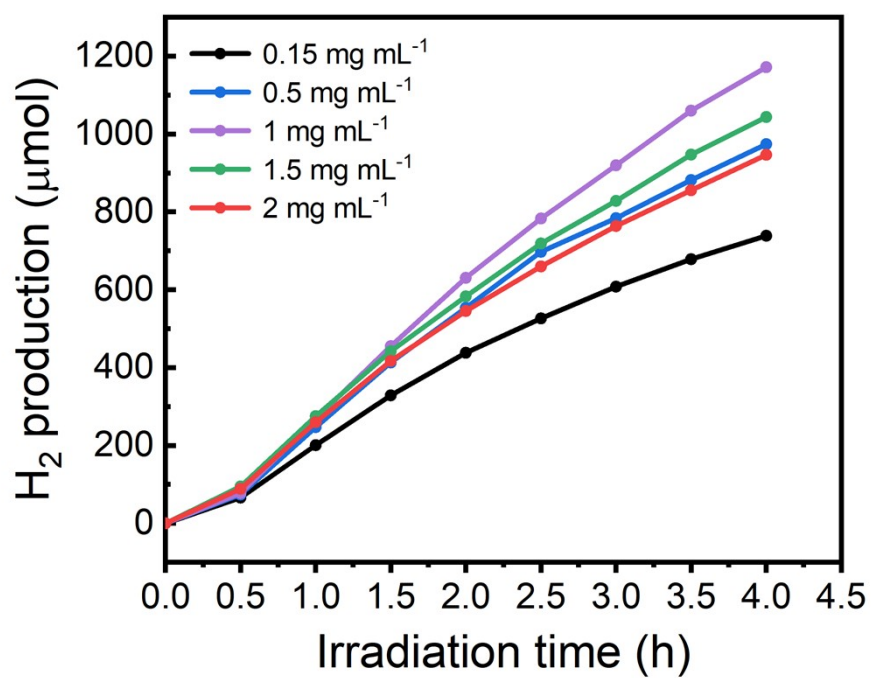
**Fig. S2** The photographs of ice block in stainless-steel cup (a) and laser scanning process (b).



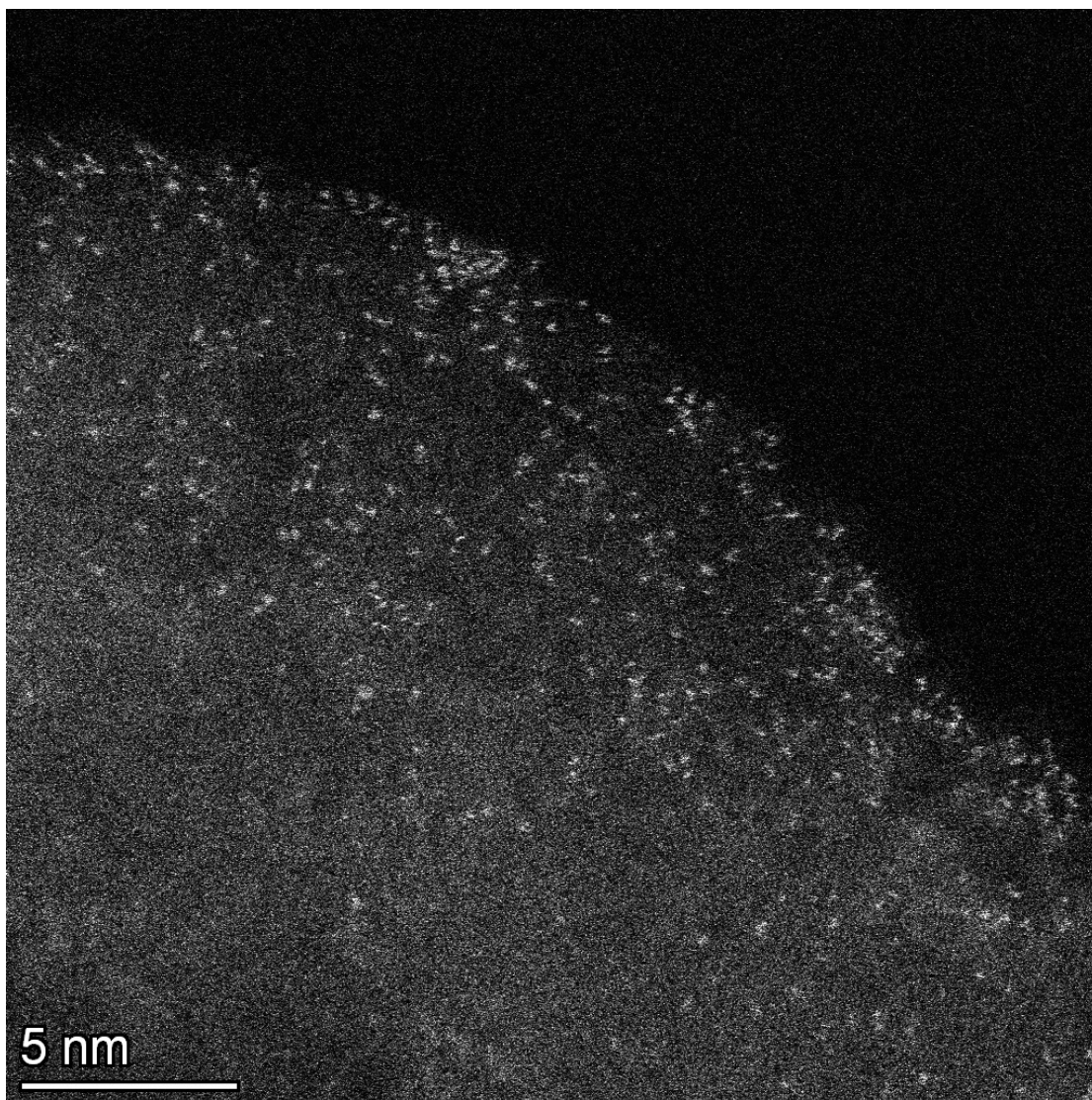
**Fig. S3** The absorbance of the ice blocks with different  $\text{H}_2\text{PtCl}_6$  concentrations.



**Fig. S4** The enlarged view of EXNES spectra.



**Fig. S5** The photocatalytic hydrogen production of Pt/g-C<sub>3</sub>N<sub>4</sub> with different H<sub>2</sub>PtCl<sub>6</sub> concentration.



**Fig. S6** The HAADF-STEM image of Pt/g-C<sub>3</sub>N<sub>4</sub> after use.

**Table S1.** The precise fitting parameters of the EXAFS result.

Sample	Bond type	CN	R (Å)	$\sigma^2$ ( $10^{-3}\text{\AA}^2$ )	R factor
Pt/g-C <sub>3</sub> N <sub>4</sub>	Pt-N	3.8±0.2	2.25±0.01	2.3±1.6	0.012
Pt foil	Pt-Pt	12	2.75±0.01	6.7±2.2	0.006
PtO <sub>2</sub>	Pt-O	5.9±1.2	2.03±0.01	1.9±1.6	0.011

CN: coordination number.  $S_0^2$  was fixed to be 0.88 from Pt foil.

$\sigma^2$ : Debye–Waller factors