

Supplementary Materials

In situ Pt single-atom trapping on TiO₂ nanotubes via ultrasonication: A one-pot approach to produce active electrodes for electrocatalytic H₂ evolution

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Table S1. Equivalent circuit parameters were obtained by fitting the impedance spectra of anatase, 2 h, and 8 h sonicated samples.

	R_s	R_{ct}	Q_{dl}	n
Anatase TNTs	2.2	93.31k	0.4×10^{-3}	0.96
2 h sonicated anatase TNTs	2.4	18.25k	1.44×10^{-3}	0.98
8 h sonicated anatase TNTs	2.3	0.95k	3.11×10^{-3}	0.95

Table S2. A comparison of the proposed HER electrocatalyst with some recently reported ones.

Electrocatalyst	Solution	Overpotential	Tafel slope (mV/decade)	Reference
Ag@Pt icosahedral NCs	0.5 M H ₂ SO ₄	35 mV @ 10 mA/cm ²	38.29	[42]
Pt-W₁₈O₄₉	0.5 M H ₂ SO ₄	743 mV @ 500 mA/cm ²	74	[43]
Mo₂N-Mo₂C/N-CW	0.5 M H ₂ SO ₄	311 mV @ 500 mA/cm ²	94	[44]
Cr-CoP-NR/CC	0.5 M H ₂ SO ₄	209 mV @ 500 mA/cm ²	44.8	[45]
Pd₃Ru/C	1 M KOH	104 mV @ 10 mA/cm ²	-	[46]
PdRu@FeO_x	1 M KOH	27 mV @ 100 mA/cm ²	83.7	[47]
Pt SAs@TNTs	0.5 M H ₂ SO ₄	116 mV @ 10 mA/cm ²	44	Present work

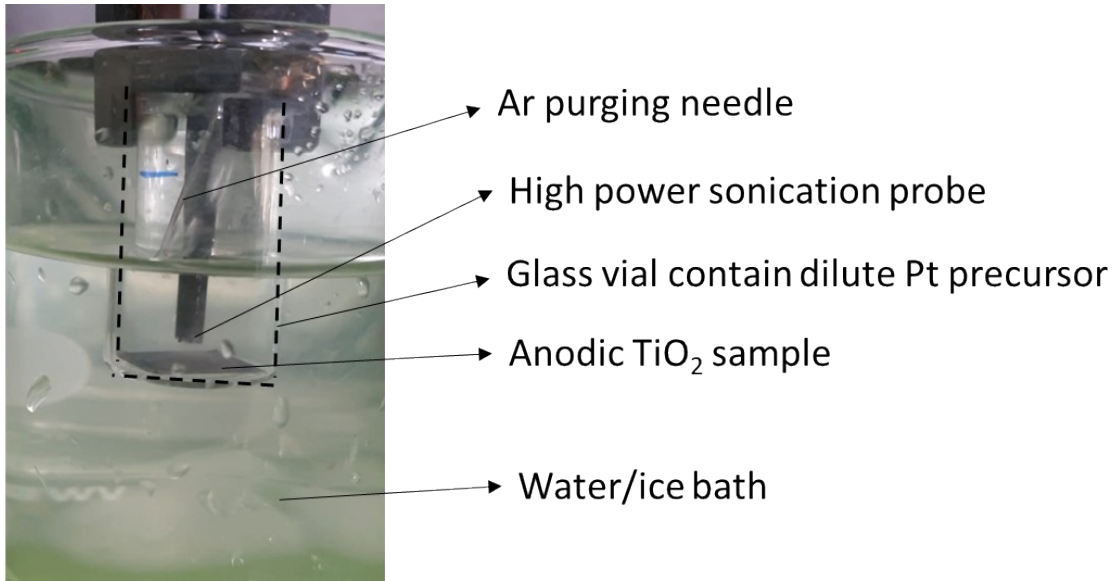


Fig. S1 Actual image of the high power sonication setup used for in situ defect formation and SA decoration of the anodic TiO₂ layer.

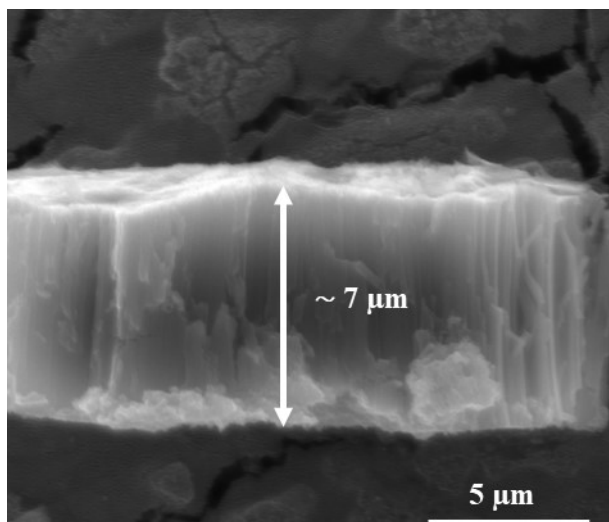


Fig. S2 SEM image of the cross-section of the TiO₂ nanotubes used in the ultrasonication process.

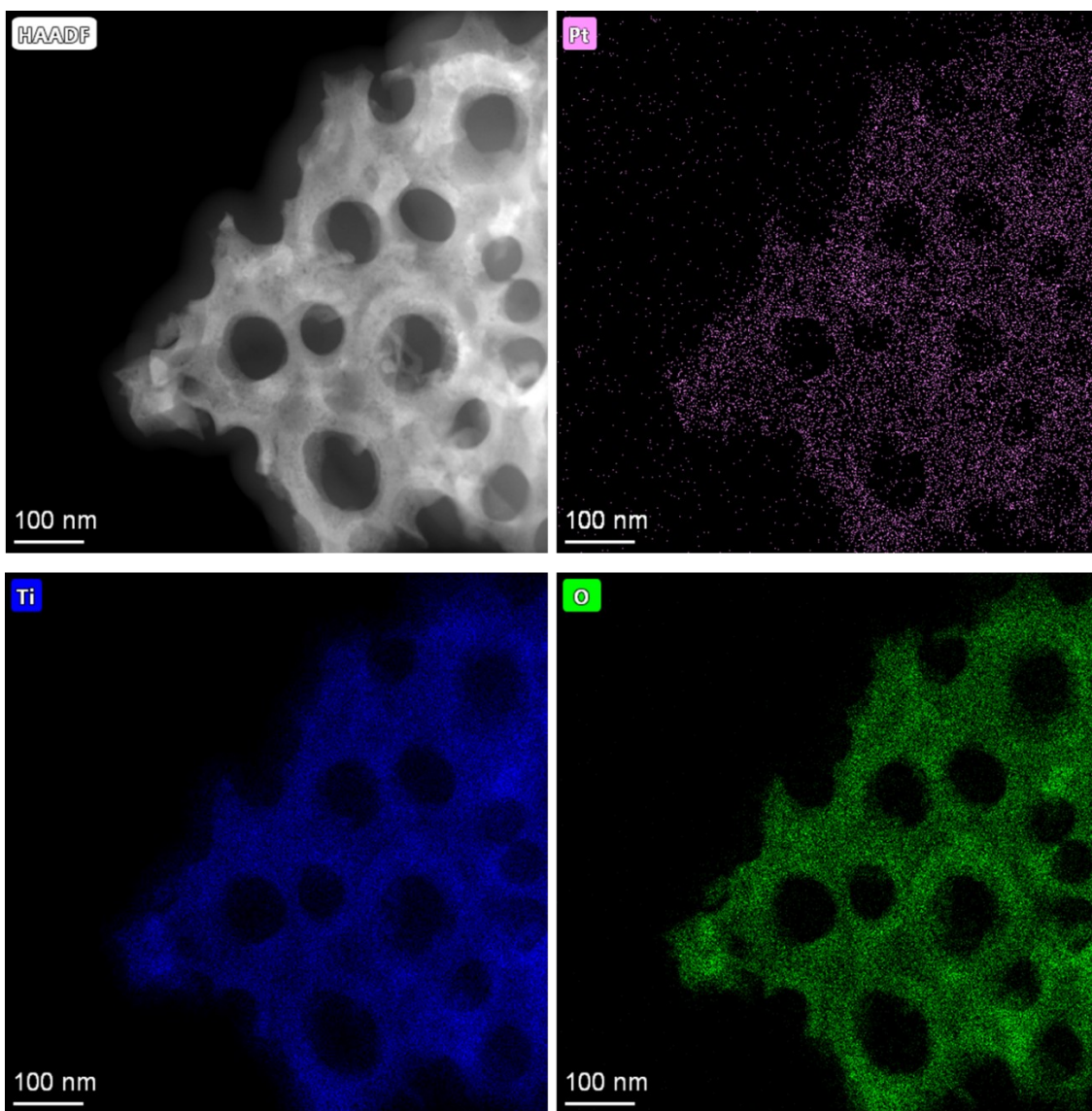


Fig. S3 HAADF-STEM image and EDX chemical maps of the top surface of the Pt-decorated TNTs sample after 8 h sonication in the Pt precursor.

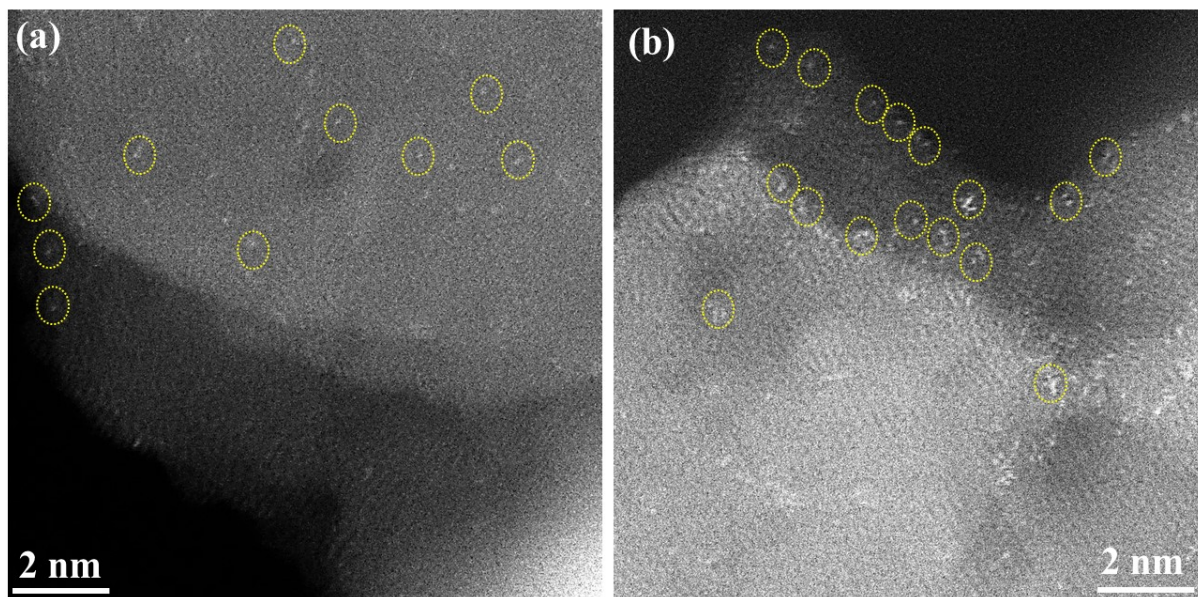


Fig. S4 HAADF-STEM images taken from the various middle-broken regions of TNTs sonicated in the diluted Pt precursor, revealing the Pt SAs decoration.

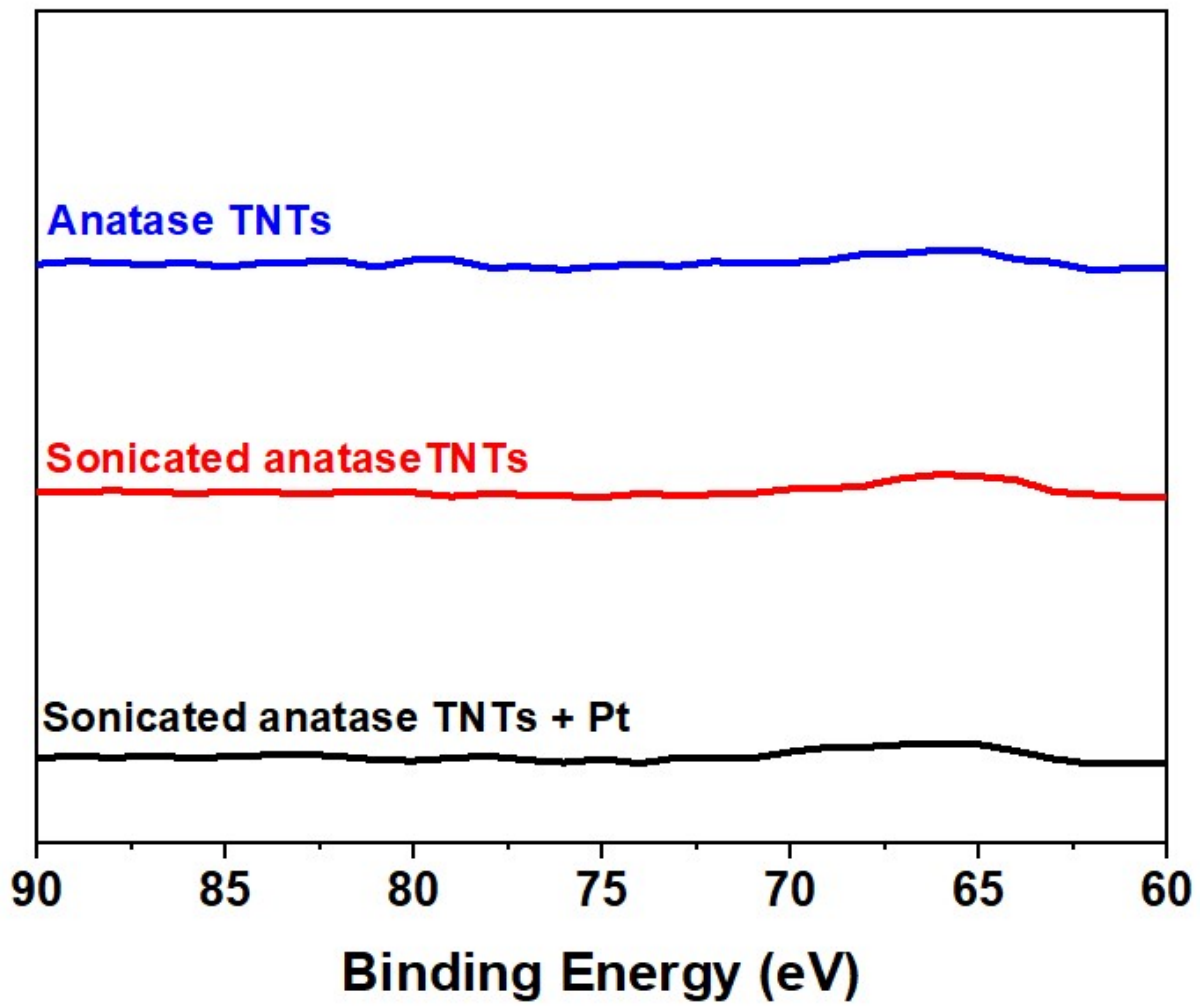


Fig. S5 A zoomed-in view of the binding energy range related to the oxidation states of Pt in the survey XPS spectrum.

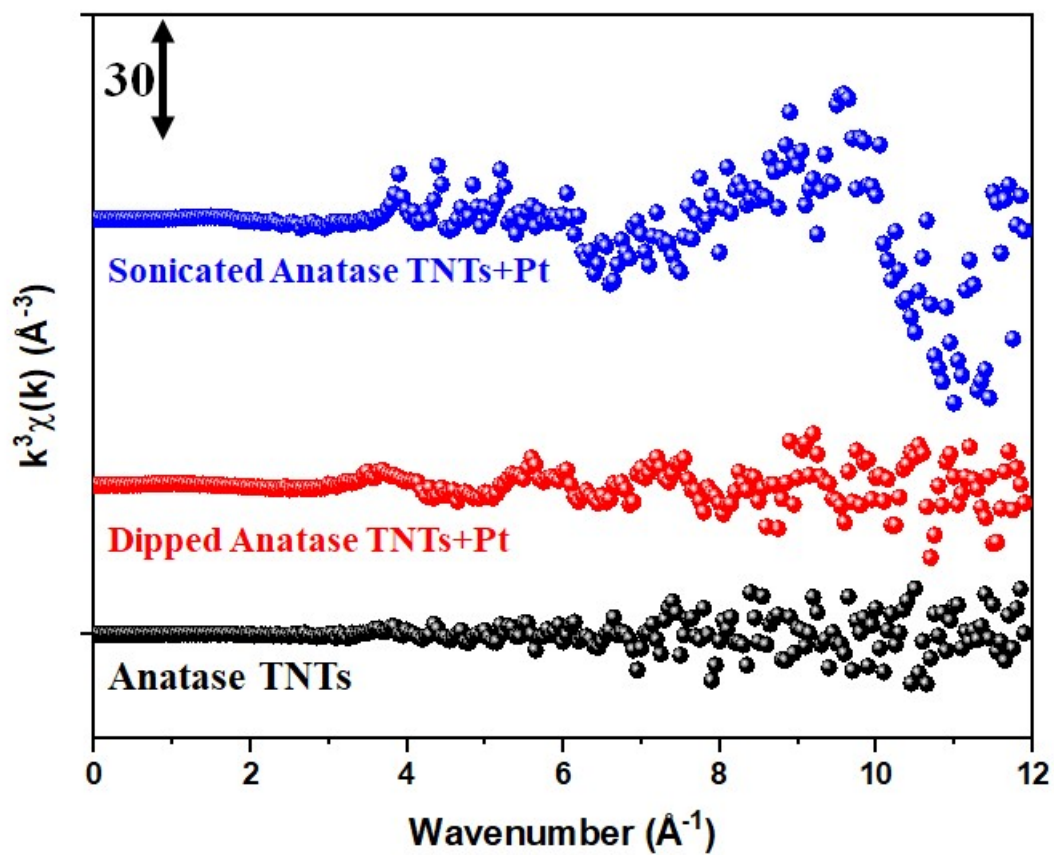


Fig. S6 K-space spectra regarding the XAS data for the anatase TiO_2 , dipped and sonicated TiO_2 nanotubes in Pt precursor with the same concentration and for the same time.

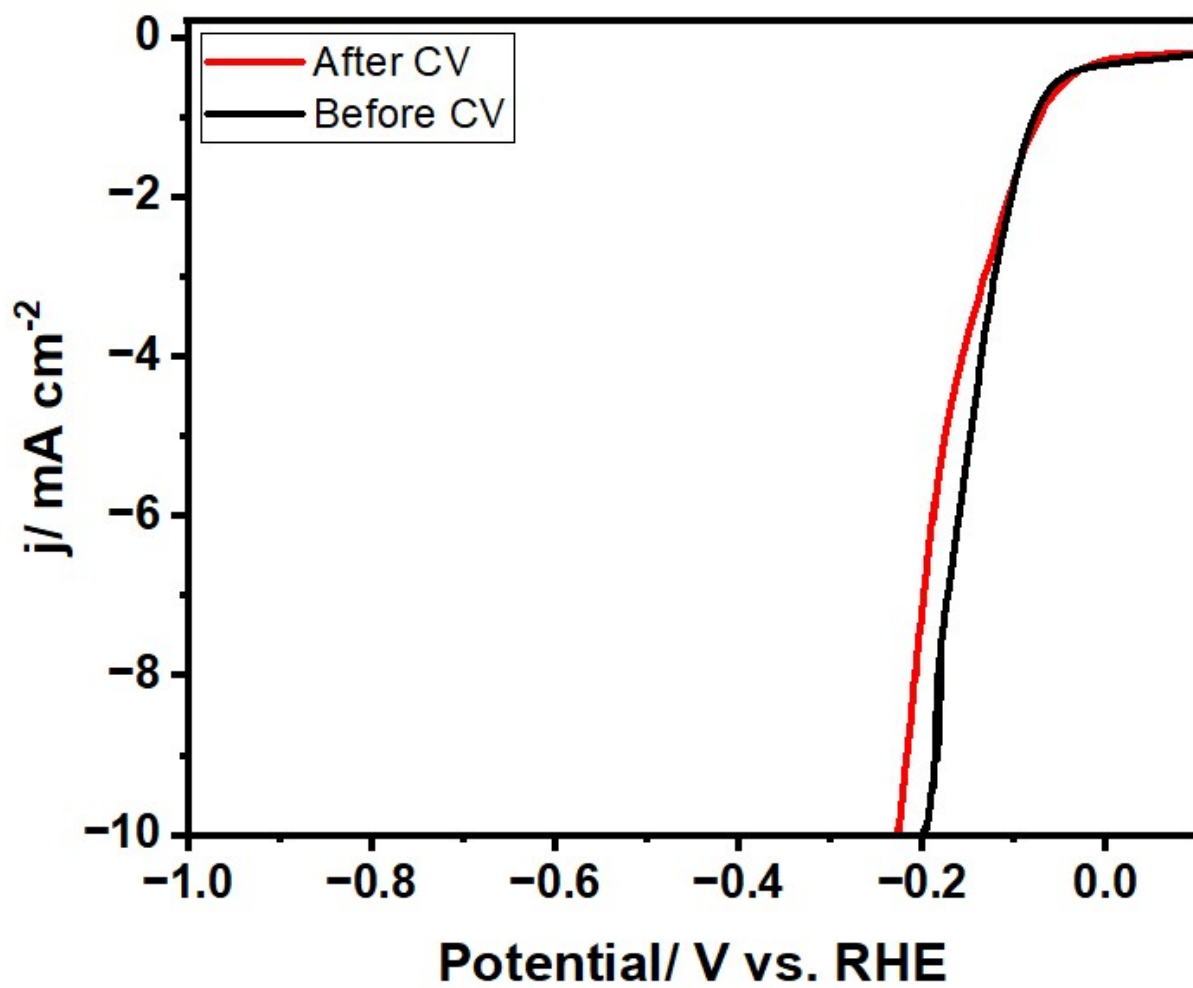


Fig. S7 LSV curves of the TNTs sonicated in Pt precursor, recorded before and after 20000 CV cycles.