

Supporting Information

Unveiling Nucleation and Evolution of Twinned Intermetallic Nanocrystals for CO-Tolerant Selective Hydrogenation

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Supplementary materials and methods

Preparation of polyoxometalates

The $\text{K}_2[\text{H}_6\text{PtMo}_6\text{O}_{24}]$ was synthesized according to the previous literature (1). Typically, 122.4 mg $\text{K}_4\text{Pt}(\text{OH})_6$ and 466.4 mg K_2MoO_4 were dissolved in 50 mL and 40 mL deionized water, respectively. Then the solution of $\text{K}_4\text{Pt}(\text{OH})_6$ and K_2MoO_4 were mixed under stirring, and the pH value of the mixture was adjusted to 5.4 by 1.0 mol/L HNO_3 . The as-prepared solution was evaporated to 10 mL to obtain orange-yellow crystal, then the obtained suspension was placed in refrigerator overnight. The $\text{K}_2[\text{H}_6\text{PtMo}_6\text{O}_{24}]$ powder was acquired by separated from suspension and dried in vacuum oven at 40 °C.

Supplementary results

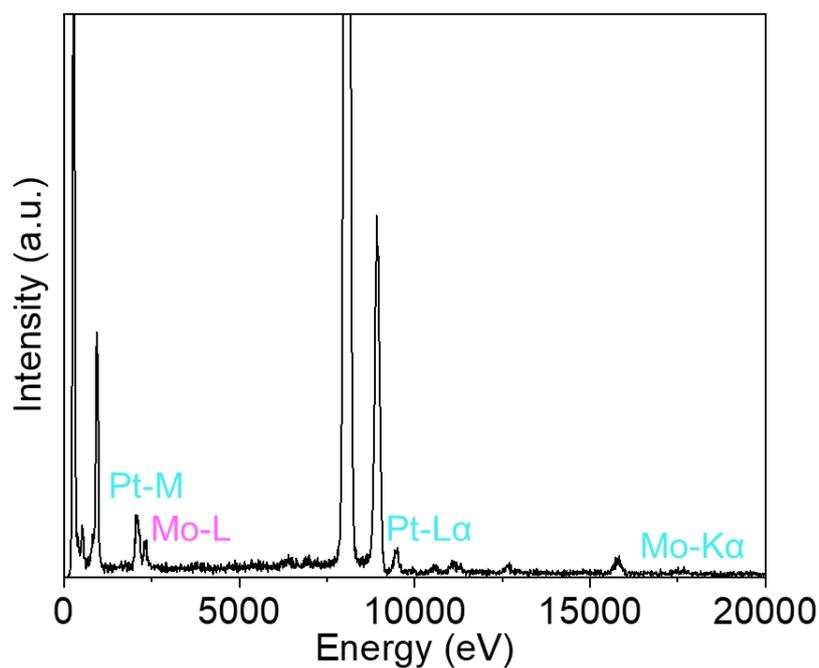


Fig. S1. The EDX spectrum of an individual intermetallic Pt_2Mo nanocrystal prepared under a heating rate of 25 °C/min and annealing at 1000 °C for 4 h.

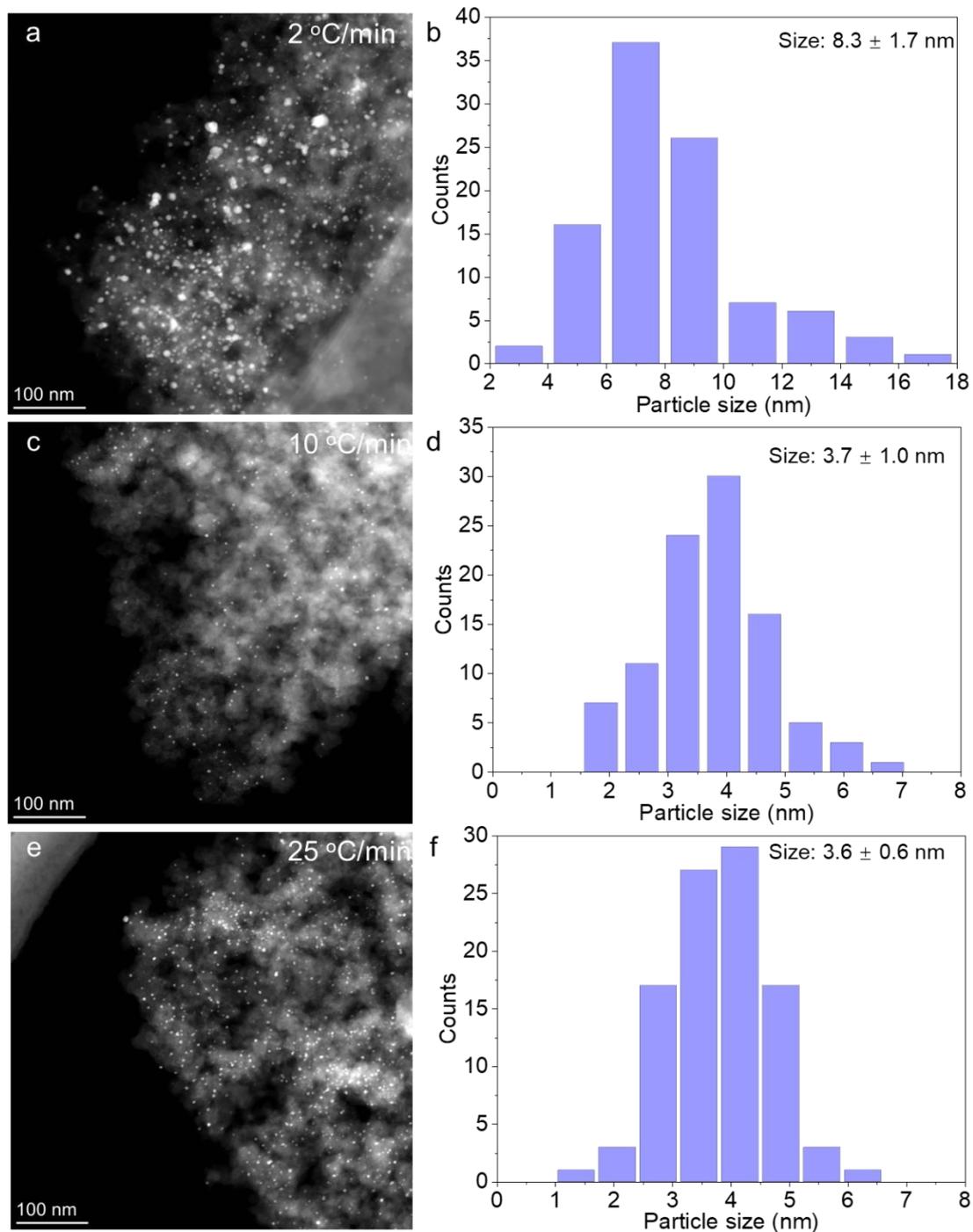


Fig. S2. HADF-STEM images and particle size distributions of Pt₂Mo nanoparticles prepared under different heating rates: 2 °C/min (a, b), 10 °C/min (c, d), 25 °C/min (e, f). The heating time at 1000 °C is 4 h.

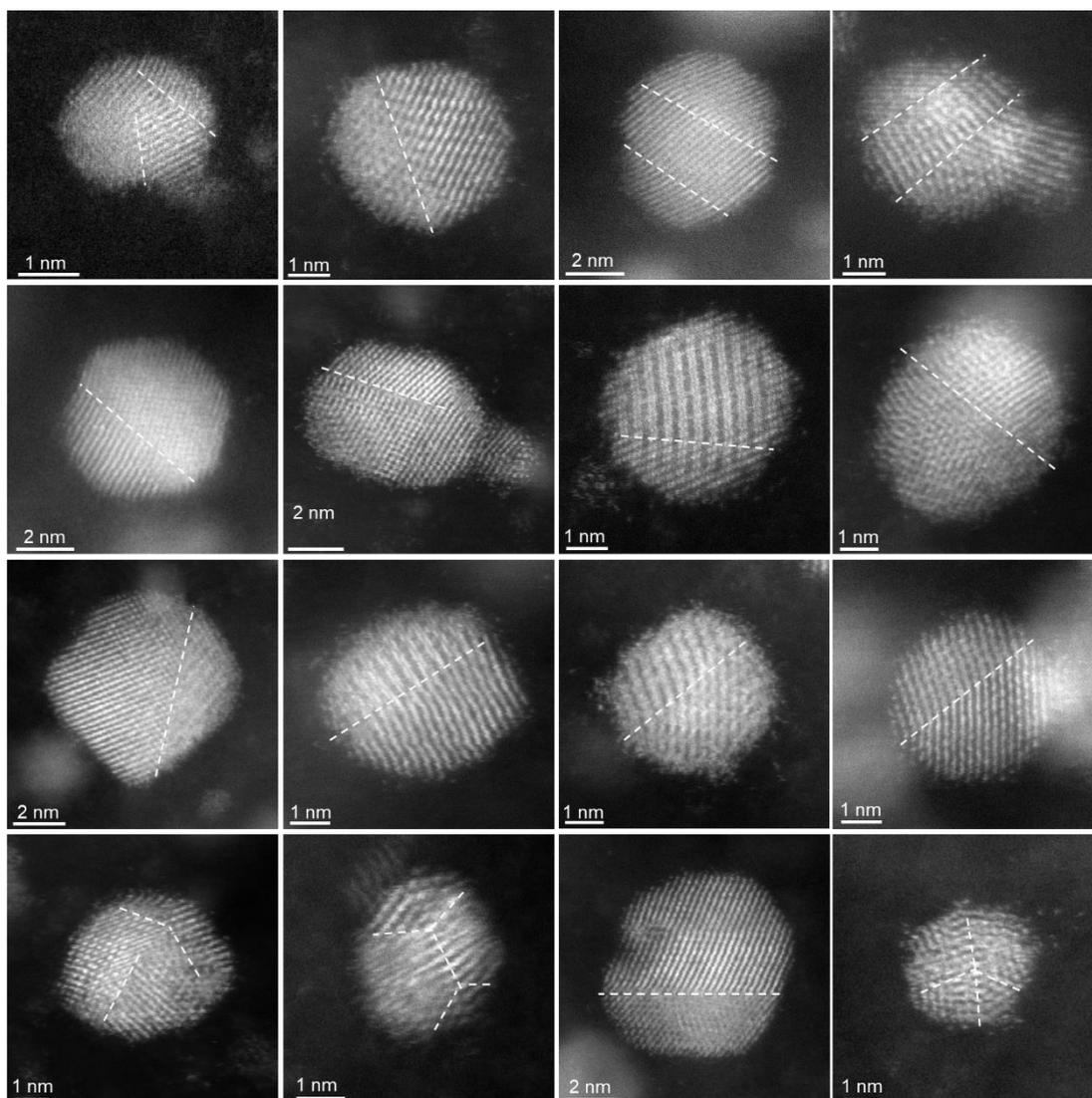


Fig. S3. HAADF-STEM images of twinned Pt₂Mo/C prepared at 1000 °C under a heating rate of 25 °C/min. The twin boundary is marked by dashed line.

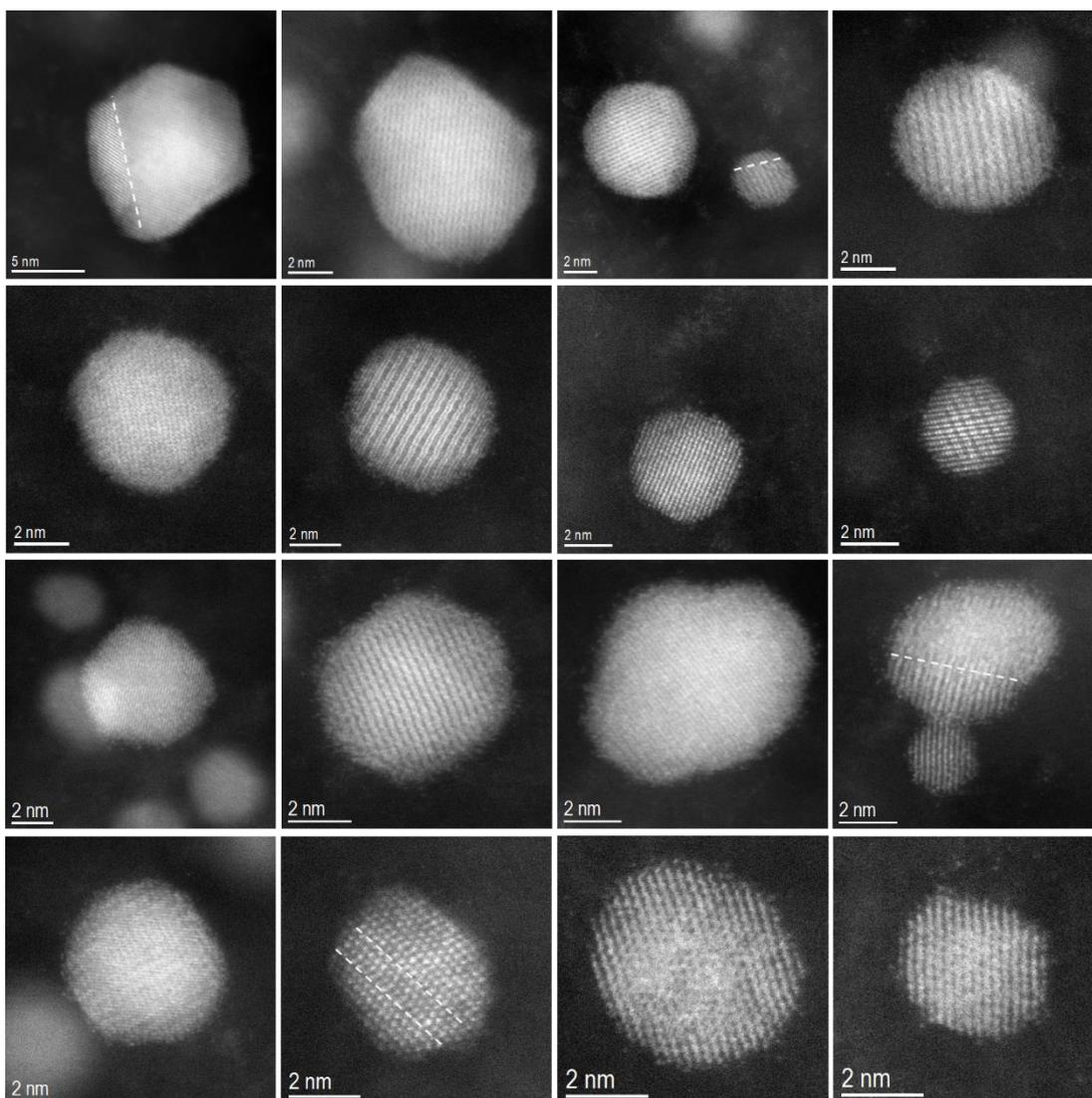


Fig. S4. HAADF-STEM images of Pt₂Mo/C prepared at 1000 °C under a heating rate of 2 °C/min. The twin boundary is marked by dashed line.

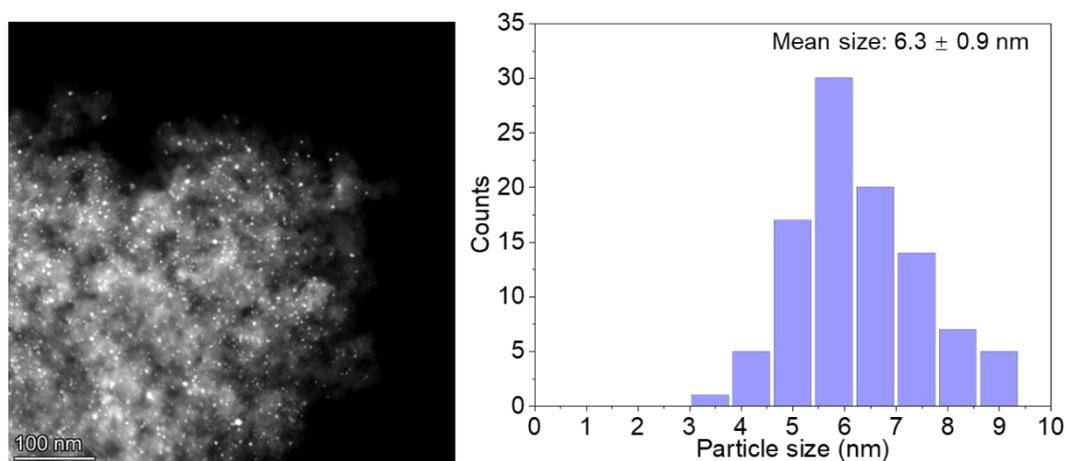


Fig. S5. HAADF-STEM image and particle size distribution of Pt₂Mo/C prepared under a heating rate of 25 °C/min and annealed at 1000 °C for 8 h.

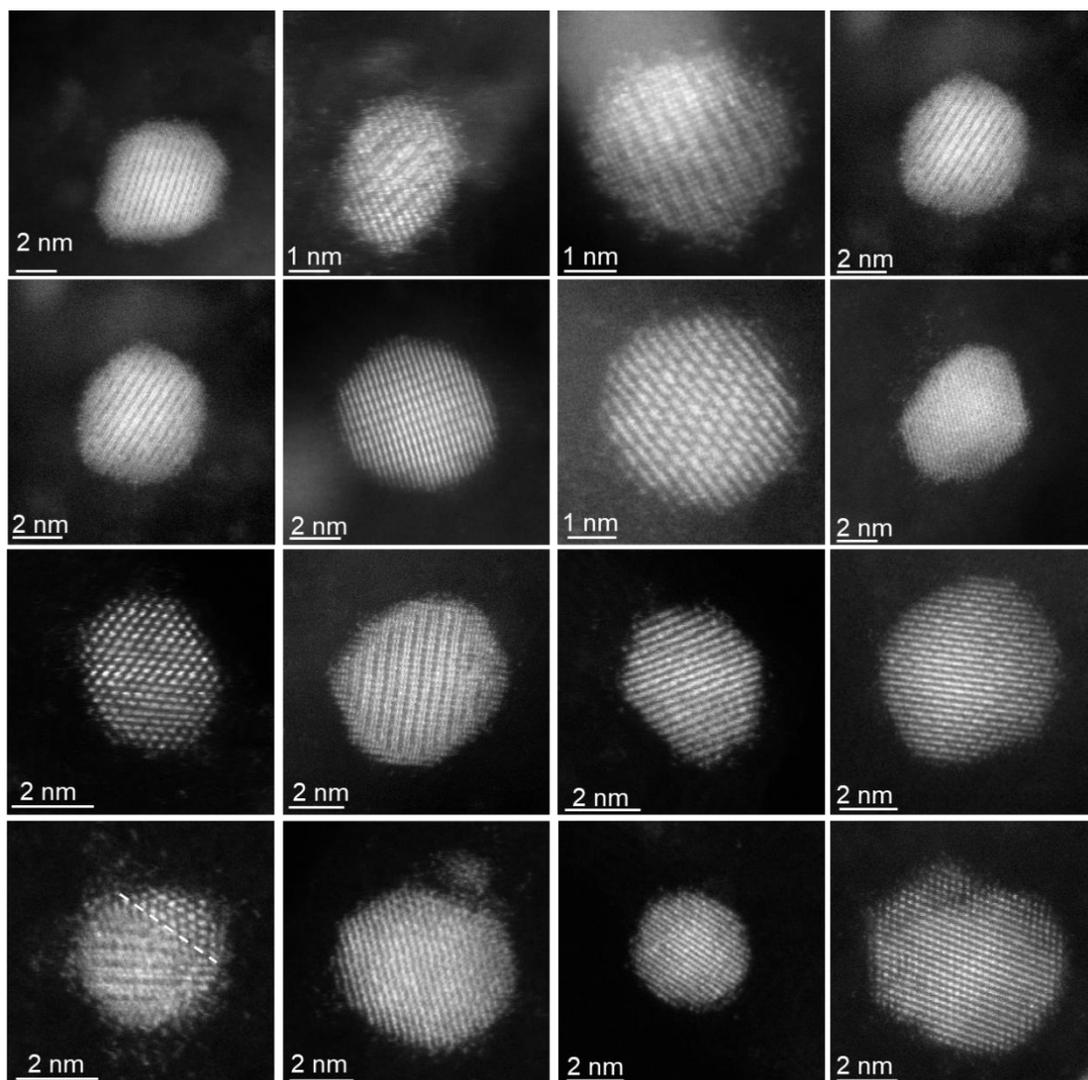


Fig. S6. STEM characterizations of twinned Pt₂Mo/C (25 °C/min, 8 h) prepared at 1000 °C.

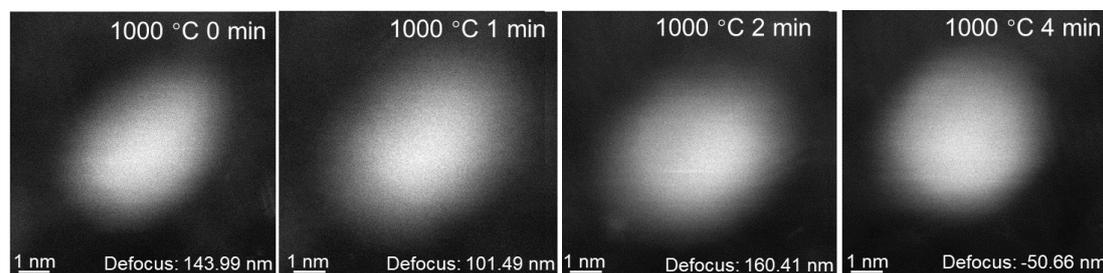


Fig. S7. Time-sequenced HAADF-STEM images of a molten Pt₂Mo nanoparticle at 1000 °C. The images were acquired with tuning the e-beam defocus continuously.

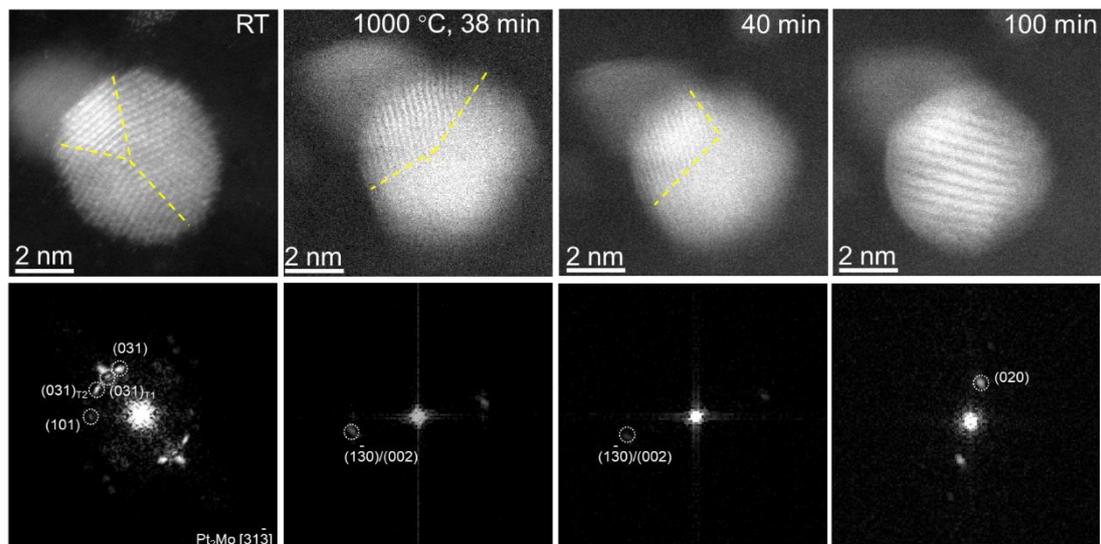


Fig. S8. In-situ STEM observation of the conversion of twinned Pt_2Mo to untwinned Pt_2Mo nanocrystals at $1000\text{ }^\circ\text{C}$.

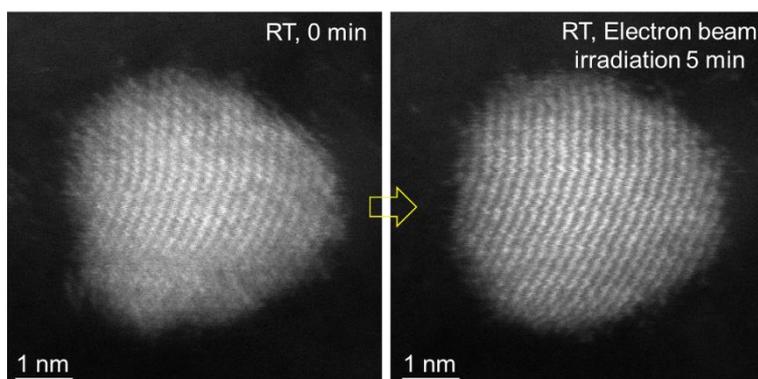


Fig. S9. The HAADF-STEM images of twinned Pt_2Mo nanocrystal before and after electron beam irradiation for 5 min at $25\text{ }^\circ\text{C}$. Conditions: acceleration voltage of 300 kV, screen current of 0.1 nA, convergence angle of 25 mrad, and the probe area of 1 nm^2 .