

Tension-strained mesoporous Pt nanosheets for Li-CO₂ battery with low overpotential

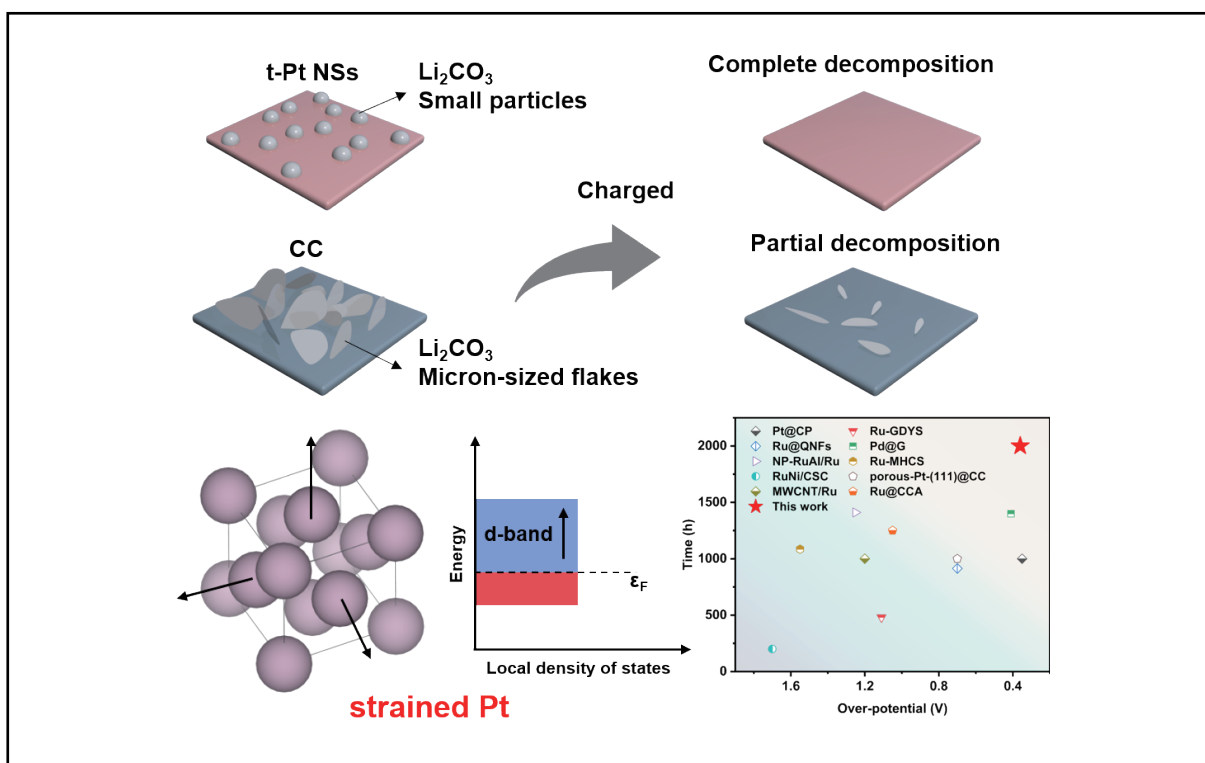
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Graphical abstract



Pt nanosheet catalyst with tensile strain increases Li-CO₂ battery performance.

Public summary

- By modulating the electronic structure and upshifting the d-band center of Pt nanosheets, tensile strain enhances their intrinsic catalytic activity, thereby enabling efficient CO₂ reduction and Li₂CO₃ decomposition, leading to significantly improved electrochemical performance in Li-CO₂ batteries.
- Mesoporous Pt nanosheets with high specific surface area provide more active sites for the catalytic reaction and promote the adsorption of CO₂ and Li₂CO₃, thus greatly improving the kinetics of the CO₂ reduction reaction and CO₂ evolution reaction processes.
- With a low overpotential of 0.36 V sustained over 2000 h at 10 μA·cm⁻² and 100 μAh·cm⁻², the tensile strain mesoporous Pt nanosheets pave the way for rationally designing strain-engineered metal-based materials in electrochemical energy storage.

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Abstract: The slow kinetics of the cathode CO₂ reduction reaction and the decomposition reaction of Li₂CO₃, a wide-bandwidth insulating product, lead to difficult CO₂ capture and high charging potential in Li–CO₂ batteries. To improve the reaction kinetics and decrease the reaction overpotential, we synthesized mesoporous Pt nanosheets with high tensile strain. The presence of many unsaturated coordinated Pt atoms around the pores gives rise to tensile strain in the mesoporous Pt nanosheets. This tensile strain plays a key role in regulating the interactions between the catalytic surface of Pt and the adsorbed intermediates. The two-dimensional structure provides more active sites on the surface for the catalytic reactions. These superiorities enable a low overpotential of 0.36 V at a cutoff capacity of 100 μAh·cm^{−2} at a current density of 10 μA·cm^{−2} over more than 2000 h. This study opens new possibilities for the rational design of metal-based materials with strain engineering for electrochemical energy storage.

Keywords: Li–CO₂ battery; Pt nanosheets; mesoporous; tension strain

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1 Introduction

In contemporary society, the advancement of global industrialization and continuous population growth has led to the overconsumption of nonrenewable fossil fuels^[1], which has resulted not only in an energy crisis but also in the continuous emission of greenhouse gases (mainly CO₂) and accelerated global warming^[2]. Moreover, with the gradual increase in the demand for a greater energy supply, the most widely used lithium-ion batteries have gradually failed to meet the needs of generative life because of their poor energy density (350 Wh·kg^{−1}). In this context, lithium-carbon dioxide (Li–CO₂) batteries, which use carbon dioxide as a reactant, have become promising solutions because of their high theoretical energy density of 1876 Wh·kg^{−1}^[3]. However, at present, several problems, such as the large potential difference between charging and discharging and the difficulty of decomposing lithium carbonate (Li₂CO₃), which has a wide band gap, have severely restricted the development of Li–CO₂ batteries^[4].

Therefore, the development of bifunctional catalysts for the CO₂ reduction process (CRR) and CO₂ evolution process (CER) is the key to addressing the abovementioned challenges^[5]. Carbon-based catalysts, transition metal compound catalysts, single-atom catalysts, redox-mediated catalysts, and noble metal catalysts have been used in recent years to promote CO₂ reduction and Li₂CO₃ decomposition^[6]. Among them, Pt-based catalysts stand out because of their unique electronic structures and associated high activities^[7], but their overpotential and cycling stability still need further improvement. On the one hand, it is necessary to modulate the intrinsic electronic structure to increase their catalytic activity; on

the other hand, as many reactive sites should be exposed on the surface as possible to accelerate the kinetics and avoid product coverage of the surface.

Surface strain engineering is an effective strategy for modulating the electronic structure of the active site^[8]. The degree of charge overlap between metal atoms, which is closely related to the adsorption of reactants and products, can be effectively regulated by introducing strain during the synthesis process^[9]. It is well known that edge sites have many unsaturated coordination atoms with distinct electronic structures due to strain. These atoms lay the foundation for controllably adjusting the energy band structure of materials^[10]. Improving the interaction between the active center and the reaction intermediates is helpful.

To obtain more edge atoms, two-dimensional mesoporous Pt nanosheets were prepared via the salt template method. Modulation of the electronic structure of Pt was achieved by strain engineering, resulting in highly active catalysis for the CRR and CER^[11]. The presence of tension strain in the as-synthesized Pt nanosheets significantly increases the intrinsic activity of the Pt catalysts and improves the electrochemical performance of Li–CO₂ batteries by increasing the d-band center. Moreover, porosity promotes CO₂ adsorption^[12]. The CRR and CER kinetics were thus greatly improved compared with those of other Pt-based catalysts previously applied in Li–CO₂ batteries^[13]. The Li–CO₂ battery demonstrated a low overpotential (0.36 V) after a prolonged cycle life (2000 h) at 10 μA·cm^{−2} and 100 μAh·cm^{−2}. This study opens new possibilities for strain engineering for the anode design of highly efficient Li–CO₂ batteries.

2 Materials and methods

2.1 Chemicals

Acetylacetone platinum (Pt(acac)₂) was purchased from Shanghai Bider Pharmaceutical Co., Ltd., with a purity of 98%. Potassium nitrate (KNO₃) was purchased from Sino-pharm Group Chemical Reagent Co., Ltd., AR analysis pure. Nafion solution (5%) was purchased from Sigma-Aldrich. Dimethyl sulfoxide (DMSO, anhydrous) and lithium bis((tri-fluoromethyl)sulfonyl)azanide (LiTFSI, 99%) were purchased from Aladdin. 1-Methyl-2-pyrrolidinone (NMP, AR) and nitric acid (HNO₃, AR) were purchased from Sinopharm Chemical Reagent Co., Ltd. Lithium nitrate (LiNO₃, 99.9%) and amorphous boron powder (98%) were purchased from Shanghai Macklin Biochemical Co., Ltd. All the chemicals were used without any further purification.

2.2 Synthesis of mesoporous Pt nanosheets

For the synthesis of mesoporous Pt nanosheets, 10 mg Pt(acac)₂ and 20 mg KNO₃ were weighed in a mixed solution of ethanol and deionized water, and magnetic stirring fully dissolved them. The dispersed solution was dried to obtain a solid powder mixture, ground and evenly poured into a porcelain boat, placed in a tube furnace, held at 300 °C under an air atmosphere for 90 min, and then cooled to room temperature. The resulting product was repeatedly washed and centrifuged with a deionized water-ethanol mixed solution until the salt template was removed. Finally, the centrifuged samples were dried in a blast oven at 60 °C for 12 h for further characterization or application.

2.3 Characterization

The structures of the samples or cathodes were characterized via power X-ray diffraction patterns (SmartLab SE, Rigaku) and Raman spectroscopies (LabRAM HR Evolution). The surface compositions of the materials and the valence states of the elements were determined via X-ray photoelectron spectroscopy (Thermo Scientific ESCALAB 250Xi). Scanning electron microscopy (SEM) images were collected on a Gemini SEM 360 scanning electron microscope. Transmission electron microscopy (TEM) and transmission electron microscopy equipped with energy-dispersive spectroscopy (TEM-EDS) images were taken on a JEM-2100 Plus transmission electron microscope. High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) images were taken on a Themis Z spherical aberration-corrected transmission electron microscope.

2.4 Battery assembly and electrochemical measurements

The electrode slurry was prepared by mixing carbon nanotubes (CNTs), Nafion solution, and a certain amount of isopropyl alcohol. The mass ratio of CNTs to tensile strain-rich mesoporous Pt nanosheets (t-Pt NSs) was 1 : 1. The mixed paste was sprayed on a carbon cloth with a diameter of 12 mm via a spray gun. CR2032 coin cells with 19 holes were assembled in an argon-filled glovebox with water and oxygen concentrations maintained below 0.01 ppm. Whatman glass fiber separators were used, and the electrolyte consisted of 1 M LiTFSI in DMSO with the addition of 0.3 M LiNO₃.

The prepared electrodes were sealed in glass test cells filled with CO₂ for electrochemical testing. The galvanostatic charge-discharge tests were conducted using a Neware testing station, with all capacities calculated on the basis of the electrode area. Electrochemical impedance spectroscopy (EIS) in the frequency range of 100 kHz–100 mHz and cyclic voltammetry (CV) in the voltage range of 2.0–4.0 V at a scan rate of 0.4 mV·s⁻¹ were performed on a BioLogic VSP electrochemical workstation. EIS fitting was performed via ZView.

3 Results and discussion

As shown in Fig. 1a, t-Pt NSs were obtained by calcining a mixture of Pt(acac)₂ and KNO₃ at 300 °C. Sheet-like nanostructures could be clearly observed by SEM (Fig. 1b), and TEM-EDS analysis revealed that the Pt was uniformly dispersed throughout the nanosheets without the presence of other elements (Fig. 1c, d). To verify the potential of t-Pt NSs, the Brunauer-Emmett-Teller (BET) surface area was also obtained from the N₂ adsorption-desorption isotherms. The specific surface area of t-Pt NSs is 4.74 m²·g⁻¹, which provides a larger specific surface area and more active sites for the adsorption of carbon dioxide molecules during the discharge process and improves the carbon dioxide reduction kinetics and rate performance of the battery. There were many visible pores throughout the entire nanosheet (Fig. 1e, f). Most pores clearly exhibit a narrow size distribution of only approximately 3–5 nm, which is also shown in Fig. 1g^[12]. The mesoporous nature results in a large surface area and many unsaturated coordinating Pt atoms around the pores. Intensive tensile strain may occur along the pore edges.

To illuminate the possible tensile strain, detailed structural characterizations were carried out. The X-ray diffraction pattern in Fig. 2a of the t-Pt NSs matches well with the standard card (PDF#87-0646), with no other species detected, indicating the pure phase of cubic Pt (Fm-3m). Compared with that of the standard card, the (111) peak was shifted slightly toward lower angles. The calculated cell parameter *a* of 0.392 nm is larger than the standard value of 0.391 nm, indicating a possible lattice expansion^[14]. Furthermore, the selected-area electron diffraction pattern (SAED) shown in the inset of Fig. 2b was subjected to azimuthal integration to obtain the corresponding diffraction profile. The structure factors were derived by subtracting the atomic scattering factors from the distribution and then subjected to a Fourier sinusoidal transformation to produce the pair distribution function (PDF)^[15] (Fig. 2c). The spacings between the nearest neighboring Pt atoms in t-Pt increased from the theoretical value of 2.74 Å to 2.88 Å, and similarly, the spacings to their 2nd and 3rd nearest neighbors mirrored the trend. To further visualize the atomic location in the nanosheets, spherical aberration-corrected transmission electron microscopy (ACTEM) was used to confirm the presence of atomic extension. The Pt atoms on the (01-1) facet were observed carefully as a typical example. As labeled with the red line and purple line in Fig. 2d, the atomic distances increase. The atomic distance in the red line increased to 0.495 nm compared with the theoretical value of 0.426 nm, whereas the atomic distance in the

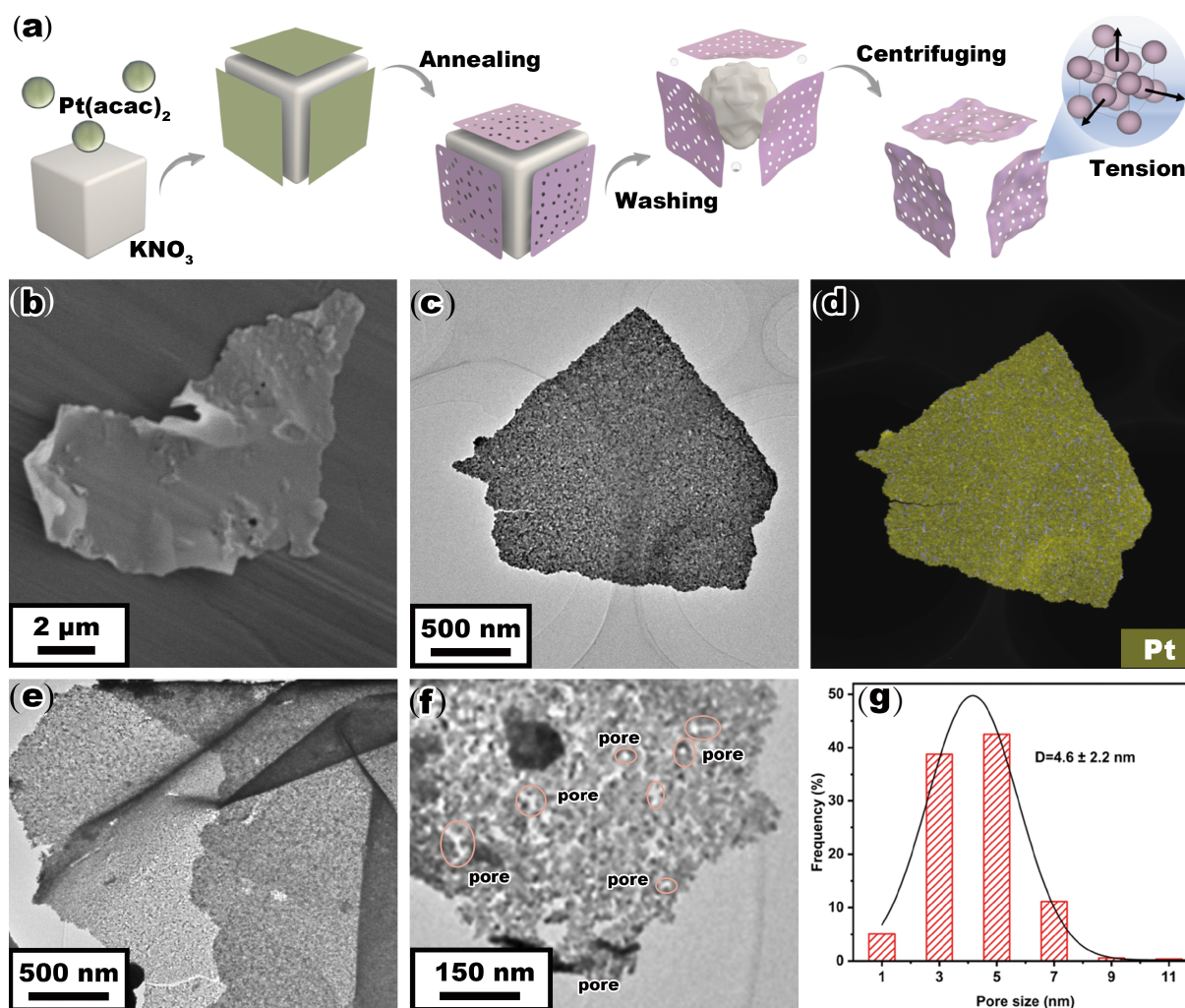


Fig. 1. Morphological characterization of t-Pt NSs. (a) Schematic diagram of the fabrication process; (b) SEM images; (c, d) TEM-EDS images; (e) TEM images at low magnification; (f) TEM images at high magnification; (g) histogram of the distribution of pore sizes in t-Pt NSs.

direction of the purple line increased from the theoretical value of 0.276 nm to 0.289 nm (Fig. 2e). The induced controlled lattice strain is approximately 16.2% and 4.7%, respectively, as determined by the following equation: $\varepsilon = (d_{\text{strain}} - d_{\text{standard}})/d_{\text{standard}}$, where d_{strain} and d_{standard} represent the strain layer spacing and the standard Pt crystallographic information file (cif) layer spacing, respectively. To verify the influence of the increase in the atomic distance, X-ray photoelectron spectroscopy (XPS) was performed to observe the change in the electronic structure (Fig. 2f). The peak of Pt 4f is shifted toward a lower binding energy, indicating a decrease in the number of ligands around the platinum and an upward shift of the d band center of the atom^[9]. Overall, as shown in Fig. 2g, it can be concluded that the atomic distance on the surface of mesoporous Pt nanosheets tends to increase with increasing microscopic tensile strain^[6]. The tensile strain results in weakened bonding between neighboring Pt atoms and elevated energy band centers of the Pt atoms. Therefore, the degree of charge overlap decreases, and the d-band center increases. Such an adjustment is favorable for subsequent CRR and CER reactions^[17]. Strain engineering is highly expected to significantly improve the electrochemical performance of Li-CO₂ battery.

To further verify the potential in Li-CO₂ batteries, for comparison, electrodes with polar sheets loaded with t-Pt NSs on the carbon cloth (CC) surface and CC were chosen to illuminate the product adsorption and decomposition ability, as shown in Fig. 3. As depicted in the XRD patterns in Fig. 3a, Li₂CO₃ formed after discharge and completely decomposed after charging in t-Pt NSs. However, for the CC substrate (Fig. 3d), Li₂CO₃ still remained and could not be completely decomposed. Raman spectroscopy further revealed the complete decomposition of the generated Li₂CO₃ on the t-Pt NSs (Fig. 3b), in agreement with the conclusion from the XRD patterns (Fig. 3a). This decomposition trend was also not observed for CC (Fig. 3e). A careful examination of the XRD patterns revealed a wider full width at half maximum (FWHM) of Li₂CO₃ for the t-Pt NS electrode, indicating a smaller grain size on the basis of the Debye-Scherrer equation. SEM analysis of the morphology of the discharged product (Fig. 3c, f) revealed that the Li₂CO₃ generated on t-Pt NSs existed predominantly in the form of small particles, increasing the susceptibility of the material to decomposition. The Li₂CO₃ produced on CC, however, were micron-sized flakes, which present greater difficulty in decomposition because of the wide-bandwidth insulating nature of Li₂CO₃.

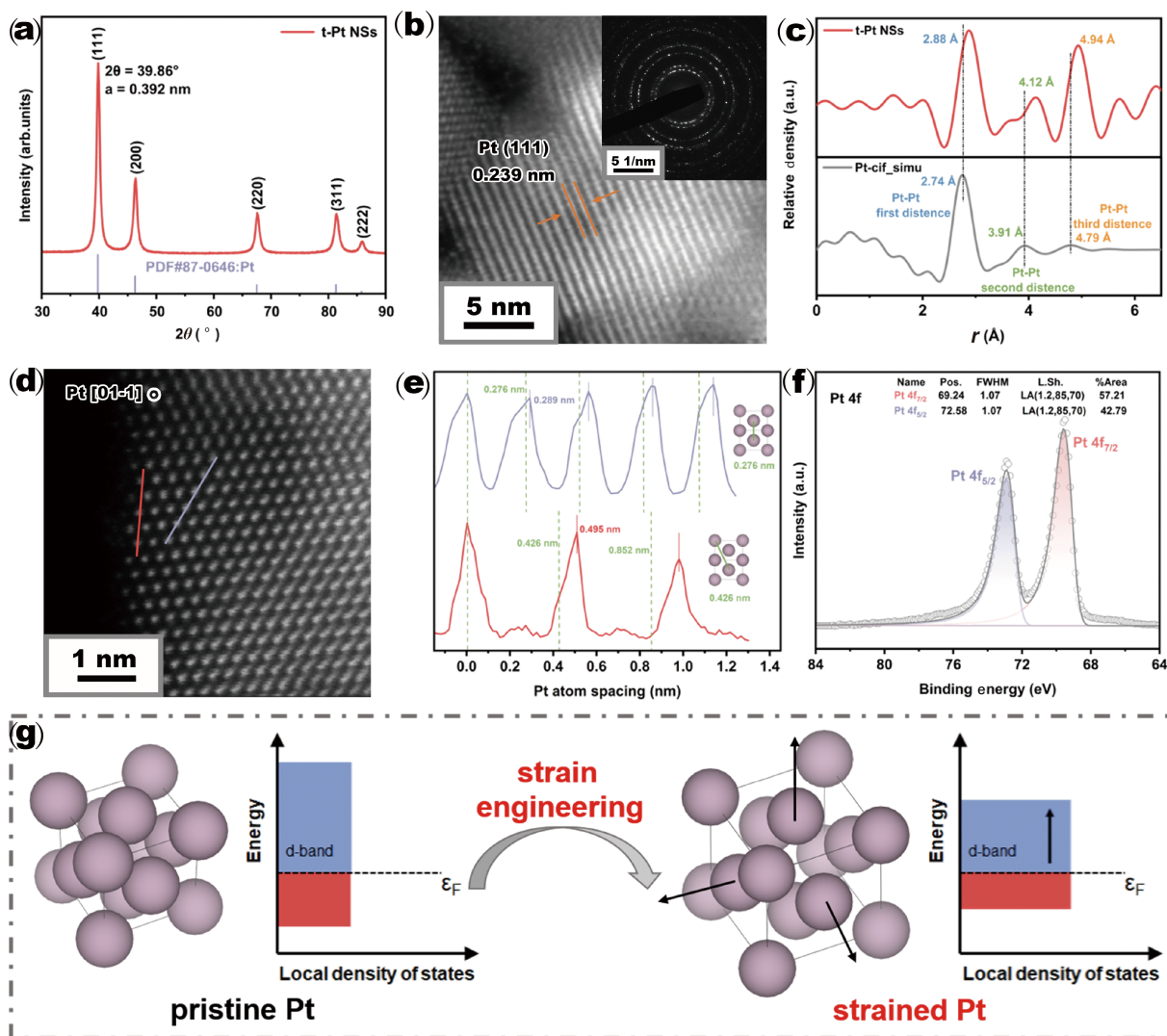


Fig. 2. (a) XRD diffraction pattern of t-Pt NSs; (b) TEM image with the inset of the corresponding SAED diffraction pattern; (c) PDF of t-Pt NSs with the simulated PDFs based on the crystalline structures of Pt; (d) atomic-level-resolved ACTEM image; (e) Pt atomic spacings versus the standard values along the labeled directions in (d); (f) Pt 4f X-ray photoelectron spectra of t-Pt NSs; (g) schematic representation of the crystal structure of Pt with the corresponding energy band structure without and with tension strain.

Overall, as shown in Fig. 3g, the micron-sized flakes of Li₂CO₃ cover almost the entire surface of the CC, acting as an insulating shell that inhibits the transport of electrons, Li⁺ and CO₂ during the subsequent electrochemical reaction. In contrast, Li₂CO₃ on t-Pt NSs has a uniform distribution of small particles, which facilitates subsequent decomposition.

The abovementioned studies showed that t-Pt NSs have good potential for both the CRR process and the CER process. On the basis of these results, comprehensive electrochemical performance tests were performed. First, CV measurements were performed at a scan rate of 0.4 mV·s⁻¹ in a voltage range of 2.0–4.0 V (Fig. 4a). The departure of t-Pt NSs may be attributed to the decomposition of the discharge product at 3.6 V, which shows a new oxidation peak at 3.3 V. This, in turn, is due to the alteration of the electronic structure of Pt due to tensile strain during the reaction, which enhances the ability of the material to bind to the reaction intermediates. The discharge kinetics were assessed through

EIS (Fig. 2a), where an equivalent circuit was employed for data fitting. The circuit elements are defined as follows: R_s reflects the ohmic resistance from the electrolyte, electrodes, current leads, and other components. R_{sei} denotes the resistance of the solid electrolyte interfacial layers, whereas C_{sei} represents their capacitance. R_{ct} and C_{dl} are attributable to the charge-transfer resistance and double-layer capacitance, respectively. W_o represents the Warburg impedance attributed to a diffusion process. Under a CO₂ atmosphere, the introduction of tension-strained Pt nanosheets resulted in a diminished R_{ct} (32.2 Ω) compared with that of CC (5979.1 Ω), indicating an increase in the charge transfer efficiency and interface optimization of the tension strain. The t-Pt NSs had a higher cathodic onset potential and a lower anodic onset potential, indicating better CRR and CER kinetics. The EIS results showed that the charge transfer resistance of t-Pt NSs was much lower than that of CC, which also implied that t-Pt NSs would greatly reduce the energy loss from the charge transfer

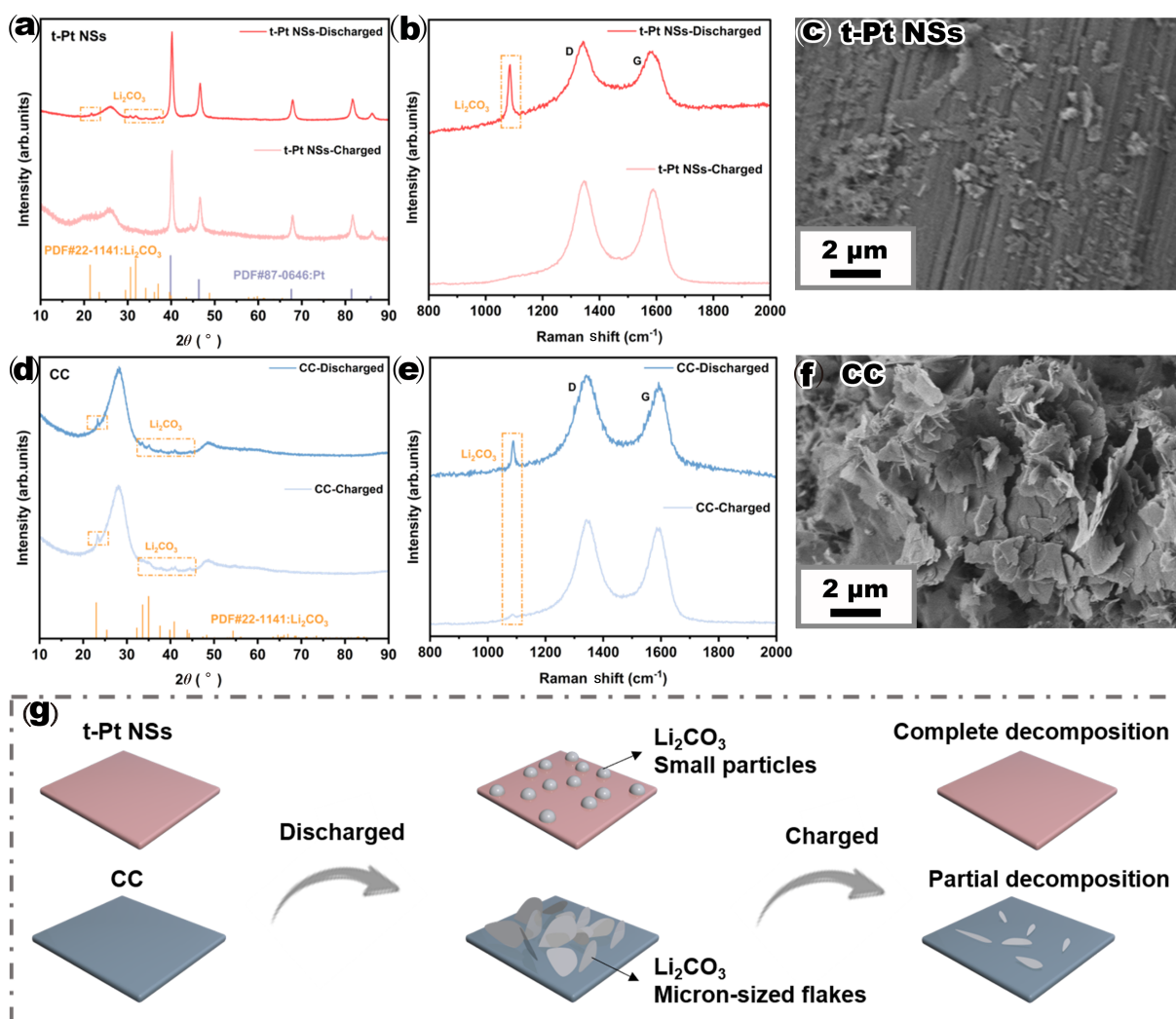


Fig. 3. (a, d) XRD diffraction patterns for t-Pt NSs and CC; (b, e) Raman spectroscopy for t-Pt NSs and CC; (c, f) SEM images of discharge products at $10 \mu\text{A}\cdot\text{cm}^{-2}$ with a limited capacity of $100 \mu\text{Ah}\cdot\text{cm}^{-2}$; (g) schematic diagram of the growth and decomposition of Li_2CO_3 on different substrates.

resistance during charging and discharging (Fig. 4b). To evaluate the effectiveness of tensile strain in accelerating the reaction kinetics, rate performance tests were performed (Fig. 4c, e). The charging voltages of 3.46 V, 3.70 V, 3.75 V, and 3.79 V for t-Pt NSs were much lower than those of 4.36 V, 4.54 V, 4.65 V, and 4.71 V for CC at current densities of $20 \mu\text{A}\cdot\text{cm}^{-2}$, $40 \mu\text{A}\cdot\text{cm}^{-2}$, $70 \mu\text{A}\cdot\text{cm}^{-2}$, and $100 \mu\text{A}\cdot\text{cm}^{-2}$, respectively. The minimum potential intervals of t-Pt NSs were 0.07 V, 0.36 V, 0.71 V, 1.48 V, 1.57 V, 1.69 V, 1.90 V, 2.13 V, and 2.37 V for different current density ranges, which were lower than the CC. After subsequent long cycles, the potential intervals were still comparable to the initial value (0.39 V) when the current density was decreased back to $10 \mu\text{A}\cdot\text{cm}^{-2}$. To better illuminate the excellent reversibility, the cycling stability at $10 \mu\text{A}\cdot\text{cm}^{-2}$ was also investigated (Fig. 4f), with a cutoff capacity of $100 \mu\text{Ah}\cdot\text{cm}^{-2}$. The t-Pt NSs maintained a small voltage gap throughout their long cycling life, and their overpotential reached only 0.36 V after 2000 h. As shown in Fig. 4g and Table 1, the low charging potential, small voltage gap and long cycling life of the t-Pt NSs are better than those in

previous reports. These results confirm that surface strain engineering is an effective strategy for optimizing the reaction kinetics, leading to the excellent electrochemical performance of t-Pt NSs in Li-CO₂ batteries.

4 Conclusions

In summary, t-Pt NSs synthesized via the salt template method were developed as advanced electrocatalysts for high-performance Li-CO₂ batteries. Tension strain was used to change the electronic structure of the metal Pt, which successfully improved its intrinsic catalytic ability and interactions with the reaction intermediates. When t-Pt NSs were used as catalysts for Li-CO₂ batteries, the batteries exhibited extremely low charge plateaus/overpotentials (3.79 V and 0.36 V) and ultralong cycle lifetimes (2000 h at a current density of $10 \mu\text{A}\cdot\text{cm}^{-2}$). Therefore, mesoporous and tension-strain-rich Pt nanosheets are favorable for the nucleation and decomposition of a wide-bandwidth insulating product, Li_2CO_3 , which improves the performance of Li-CO₂ batteries. This work not only develops high-performance Pt-based catalysts

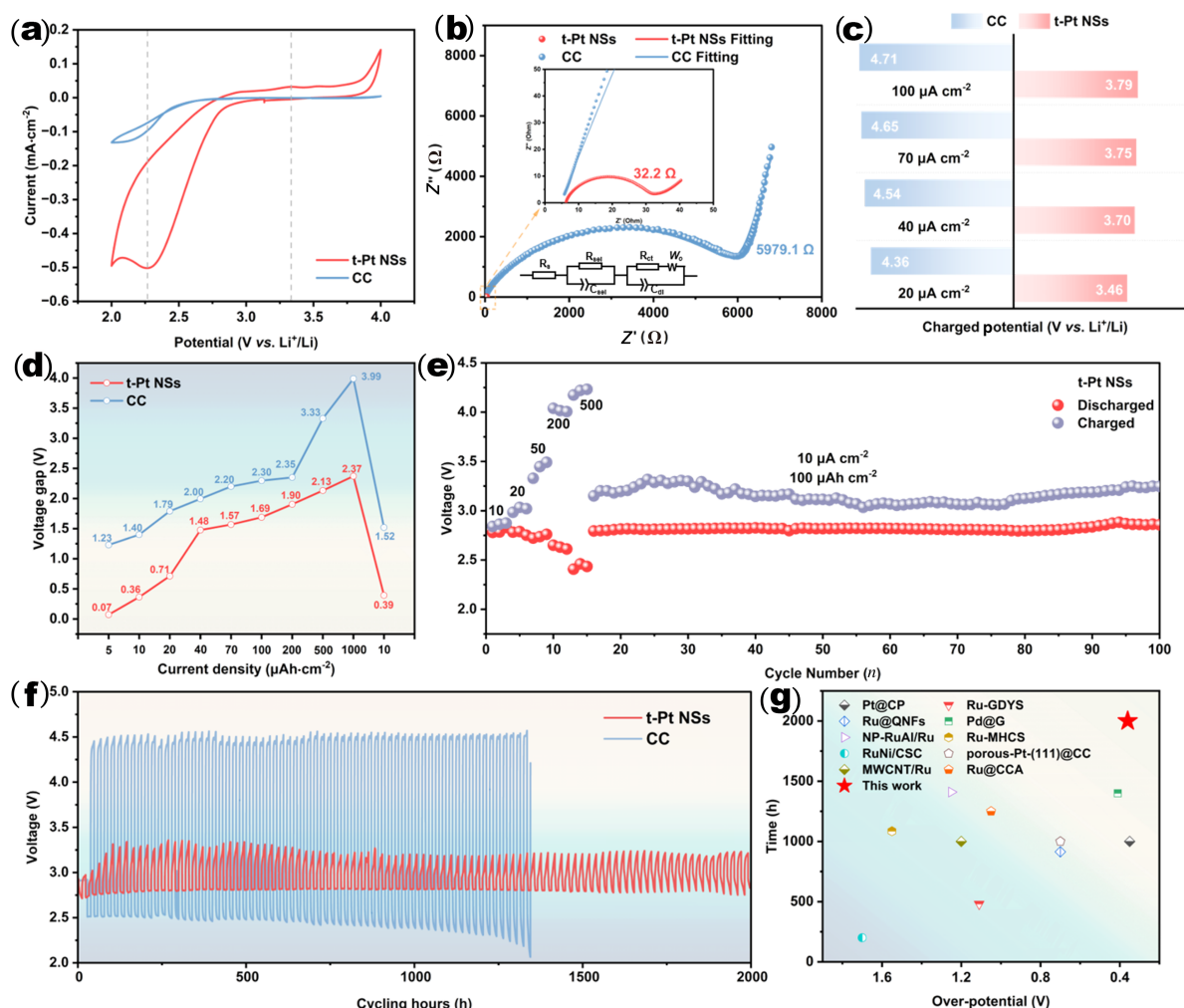


Fig. 4. Electrochemical performance of Li-CO₂ batteries with t-Pt NSs and CC. (a) CV curves in a voltage range of 2.0–4.0 V; (b) fitting of the equivalent circuit model and impedance spectrum EIS in the frequency range of 100 kHz–100 mHz; (c) charging potential at the rate of multiplication performance; (d) rate of multiplication performance; (e) cycling lap-voltage plots at different rate of t-Pt NSs; (f) long cycling performance curve at 10 μA·cm⁻², 100 μAh·cm⁻²; (g) performance comparison with the reported noble metal-based catalysts in Li-CO₂ battery.

Table 1. Comparison of the cycling performance and overpotential between t-Pt NSs and reported noble metal-based catalysts in Li-CO₂ battery.

Catalyst name	Voltage gap (V)	Cycle performance (h)	Electrolyte type	Ref.
Pt@CP	0.35	1000	1 M LiTFSI in TEGDME	[11]
Ru-GDYS	1.11	480	1 M LiTFSI in TEGDME	[18]
Ru@QNFs	0.70	914	LiNO ₃ +KNO ₃ +CsNO ₃	[19]
Pd@G	0.41	1400	1 M LiTFSI in TEGDME	[20]
NP-RuAl/Ru	1.25	1410	1 M LiTFSI in TEGDME	[21]
Ru-MHCS	1.55	1085	1 M LiTFSI in TEGDME	[22]
RuNi/CSC	1.70	200	1.0 M LiTFSI and 0.3 M LiNO ₃ in DMSO	[23]
porous-Pt-(111)@CC	0.70	1000	1 M LiTFSI in TEGDME	[13]
MWCNT/Ru	1.20	1000	Li _{1.4} Al _{0.4} Ti _{1.6} (PO ₄) ₃ (LATP)	[24]
Ru@CCA	1.05	1250	QSPE	[25]
t-Pt NSs	0.36	2000	1.0 M LiTFSI and 0.3 M LiNO ₃ in DMSO	This work

for Li-CO₂ battery, but also introduces novel opportunities for the systematic design of mesoporous two-dimensional metal-based materials, incorporating tension strain engineering, to achieve superior performance in electrochemical energy

conversion and storage.

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Conflict of interest

The authors declare that they have no conflict of interest.

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