Supplementary information

Bimetallic PtIr Nanoalloy on TiO₂-based Solid Solution Oxide with Enhanced

Oxygen Reduction and Ethanol Electro-Oxidation Performance in Direct Ethanol

Fuel Cells

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1. Experimental Section

1.1. Preparation for the mesoporous $Ti_{0.7}W_{0.3}O_2$ nanosupport

The mesoporous $Ti_{0.7}W_{0.3}O_2$ nanosupport was prepared via a one-step solvothermal process without utilizing any surfactant or further heat treatment ¹. Firstly, 0.238 mg of tungsten (VI) chloride (WCl₆, 99.9%, Sigma-Aldrich, USA) was dissolved into 50 mL of ethanol absolute (C₂H₅OH, 99.9%, Merck, Belgium) for 30 min. Next, 0.155 mL titanium (IV) chloride (TiCl₄, 99.5%, Aladdin, China) was added to the solution. Afterward, the mixture was dropped into a Teflon-lined autoclave and then transferred to an oven in which the reaction proceeded at 200 °C for 10 h. Next, the as-prepared suspension was washed with acetone (CH₃COCH₃, 99.9%, Merck, Belgium) and purified water, and the resulting product was dried at 80 °C for analysis.

2. Results and Discussion

2.1. Characterization of the mesoporous Ti_{0.7}W_{0.3}O₂ nanosupport

The crystal structure of the non-carbon $Ti_{0.7}W_{0.3}O_2$ catalyst nanosupport was determined by X-ray diffraction analysis. As shown in Fig. S1, the as-obtained $Ti_{0.7}W_{0.3}O_2$ nanosupport exhibited the anatase-TiO₂ structures (JCPDS 84-1286) with the typical diffraction peaks at 25.3°; 38.1°; 47.5°; 54.4° and 62.8° corresponding to (101); (004); (200); (105) and (204) crystal facets. No typical diffraction peaks of tungsten oxide (JCPDS 020-1324) or the segregation of W and TiO₂ were detected in the XRD profile. Furthermore, the diffraction peak of the (101) crystal facet was negatively shifted compared to the undoped TiO₂ and the standard XRD pattern of anatase-TiO₂ structure (JCPDS 84-1286), suggesting incorporation of W into the anatase-TiO₂ structure.



Figure S1. XRD profile of the non-carbon $Ti_{0.7}W_{0.3}O_2$ nanosupport in the 2 θ range from 20° to 80° at a step size of 0.02°.

The surface compositions and chemical state of the non-carbon $Ti_{0.7}W_{0.3}O_2$ nanosupport were investigated by XPS analysis, as illustrated in Fig. S2. The Ti 2p spectrum of the $Ti_{0.7}W_{0.3}O_2$ nanosupport was deconvoluted to doublets peaks at 464.5 for Ti $2p_{1/2}$ and 458.75 eV for Ti $2p_{3/2}$ of Ti(4) states, as shown in Fig. S2(a). These peaks were shifted to slightly higher binding energies than those of the undoped TiO₂ (464.0 for Ti $2p_{1/2}$ and 458.4 eV for Ti $2p_{3/2}$)². In addition, the W 4f spectrum of the $Ti_{0.7}W_{0.3}O_2$ nanosupport was deconvoluted into components of W(6) and W(4) states (Fig. S2b), suggesting the co-existence of W(6) and W(4) in the mesoporous $Ti_{0.7}W_{0.3}O_2$ nanosupport. Furthermore, the W 4f_{5/2} and W 4f_{7/2} peaks of the anatase $Ti_{0.7}W_{0.3}O_2$ nanosupport was observed at 37.25 and 35.15 eV, respectively, and was lower than that of pure WO₃ (37.4 eV for W 4f_{5/2} and 35.3 eV for W 4f_{7/2}) ^{3,4}. These results indicate the successful incorporation of tungsten into the anatase-TiO₂ structures.



Figure S2. High-resolution of (a) Ti 2p and (b) W 4f spectrums of the Ti_{0.7}W_{0.3}O₂ nanosupport.

Representative SEM and TEM images show the spherical-like morphology of the as-obtained $Ti_{0.7}W_{0.3}O_2$ nanosupports with a particle size of approximately 9 nm (Fig. S3a and b). The HR-TEM image (Fig. S3b; inset) exhibited well-defined fringes at ~3.4 Å that correspond to the spacing of the (101) crystal facet of the anatase-TiO₂, which consistent with the XRD patterns (Fig. S1). Furthermore, the XRF results (Fig. S3c) indicate that the proportion of Ti and W was 70.67 and 29.33, respectively, which is close to the theoretical ratio (Ti: W = 70: 30). The elemental mapping images of the $Ti_{0.7}W_{0.3}O_2$ nanosupport are shown in Fig. S3d-e and indicate the uniform distribution of the elements in the as-obtained catalyst support.



Figure S3. (a) SEM image, (b) TEM; inset: HR-TEM images, (c) XRF spectrum, and (d-f) elemental mapping of the mesoporous $Ti_{0.7}W_{0.3}O_2$ nanosupport.

Figure. S4 shows the N₂ adsorption/desorption isotherms and pore size distribution of the as-obtained $Ti_{0.7}W_{0.3}O_2$ and undoped TiO_2 nanomaterials The $Ti_{0.7}W_{0.3}O_2$ and undoped TiO_2 catalyst support demonstrate the hysteresis loops of the type IV isotherm (Fig. S4a and c), suggesting that the as-synthesized nanosupports are the mesoporous materials with the pore size of around 3 nm. The surface area of the mesoporous $Ti_{0.7}W_{0.3}O_2$ supports was 201.48 m² g⁻¹, which is comparable to the surface area

of the carbon black ($\sim 230 \text{ m}^2 \text{ g}^{-1}$) and higher than those of other non-carbon supports reported in previous studies (Table S1).



Figure S4. (a) N_2 adsorption-desorption isotherm and (b) pore size distribution of the $Ti_{0.7}W_{0.3}O_2$ nanosupport, (c) N_2 adsorption-desorption isotherm, and (d) pore size distribution of the TiO_2 support.

The four-point probe technique was used to record the electrical conductivity of the mesoporous $Ti_{0.7}W_{0.3}O_2$ nanospport. The electrical conductivity of $Ti_{0.7}W_{0.3}O_2$ support was ~2.2x10⁻² S cm⁻¹, which was much higher than those of other non-carbon supports reported in previous studies (Table S1) and met the requirement for support materials in fuel cells ⁵.

The poor electrochemical durability of the common carbon catalyst support is a major restriction for commercial fuel cells. The durability of the $Ti_{0.7}W_{0.3}O_2$ support was recorded by a 5000-cycle accelerated durability test (ADT) in N₂-saturated 0.5 M H₂SO₄ aqueous solution at a scan rate of 25 mV s⁻¹ (Fig. S5). As a result, the $Ti_{0.7}W_{0.3}O_2$ nanosupport exhibited superior electrochemical durability under an acidic and oxidative environment. For instance, after the 5000-cycle ADT, the CV curves of the as-obtained $Ti_{0.7}W_{0.3}O_2$ nanosupport were unchanged, while a significant change was observed in the CV curves of the Vulcan XC-72 supports, and attributed to serious electrochemical corrosion of carbon material in acidic media ⁶⁻⁹ (see Fig. S5).



Figure S5. CV curves of (a) mesoporous $Ti_{0.7}W_{0.3}O_2$ nanosupport and (b) Vulcan XC-72 support under N₂-saturated 0.5 M H₂SO₄ aqueous solution at a scan rate of 25 mV s⁻¹.

Figure. S6 shows CA curves of the as-obtained $Ti_{0.7}W_{0.3}O_2$ and Vulcan XC-72 catalyst supports in N₂-saturated 10 v/v% CH₃OH/0.5 M H₂SO₄ aqueous solution at a fixed potential of 1.60 V vs. NHE for the 3600 s test. As a result, the corrosion current density of the mesoporous $Ti_{0.7}W_{0.3}O_2$ nanosupports was 0.15 μ A cm⁻², which is 12 times lower than that of the Vulcan XC-72 supports (1.76 μ A cm⁻²), suggesting superior durability of the non-carbon $Ti_{0.7}W_{0.3}O_2$ nanosupport in an oxidizing media.



Figure S6. CA curves of the catalyst support under N₂-saturated 10 v/v% CH₃OH/0.5 M H₂SO₄ aqueous solution at the fixed potential of 1.60 V vs. NHE for 3600 s.

Table S1. A comparison of the surface area and electrical conductivity between the $Ti_{0.7}W_{0.3}O_2$ and other non-carbon nanosupports.

| Preparation route | Surface area Electrical conductive | | Refs |
|--------------------|---|--|--|
| | $(m^2 g^{-1})^{(a)}$ | (S cm ⁻¹) ^(b) | 1015 |
| Solvothermal | 201.48 | 2.20x10 ⁻² | 1 |
| Solvothermal | 125.51 | 6.23x10 ⁻⁶ | 1 |
| Hydrothermal | 98.03 | 3.00x10 ⁻² | 10 |
| Sol-gel | 26.00 | 2.09x10 ⁻¹ | 11 |
| Thermal hydrolysis | 8.60 | 8.73x10 ⁻⁴ | 12 |
| Sol-gel | - | 2.00x10 ⁻² | 13 |
| Sol-gel | 46.00 | 1.40x10 ⁻³ | 14 |
| Aerogel | 140.00 | 3.00x10 ⁻¹ | 15 |
| | Preparation route Solvothermal Solvothermal Hydrothermal Sol-gel Thermal hydrolysis Sol-gel Sol-gel Aerogel | Preparation routeSurface area (m² g⁻¹)(a)Solvothermal201.48Solvothermal125.51Hydrothermal98.03Sol-gel26.00Thermal hydrolysis8.60Sol-gel-Sol-gel46.00Aerogel140.00 | Preparation routeSurface area $(m^2 g^{-1})^{(a)}$ Electrical conductivity $(S cm^{-1})^{(b)}$ Solvothermal201.482.20x10 ⁻² Solvothermal125.516.23x10 ⁻⁶ Hydrothermal98.033.00x10 ⁻² Sol-gel26.002.09x10 ⁻¹ Thermal hydrolysis8.608.73x10 ⁻⁴ Sol-gel-2.00x10 ⁻² Sol-gel46.001.40x10 ⁻³ Aerogel140.003.00x10 ⁻¹ |

^(a)Calculation from the Brunauer-Emmett-Teller (BET) method.

^(b)Calculation from the standard four-point probe.



2.2. Characterization of the bimetallic $Pt_3Ir/Ti_{0.7}W_{0.3}O_2$ electrocatalyst

Figure S7. XRD profile of the Pt_3Ir/C catalyst in the 2 θ range from 20° to 80° at a step size of 0.02°.



Figure S8. High-resolution of (a) Pt 4f and (b) Ir 4f spectrums of the bimetallic Pt₃Ir/C catalyst.



Figure S9. High-resolution of the Pt 4f spectrum of the commercial Pt/C (E-TEK) electrocatalyst.

Table S2. Summary of the XPS results of the $Pt_3Ir/Ti_{0.7}W_{0.3}O_2$, Pt_3Ir/C and commercial Pt/C (E-TEK) electrocatalysts.

| | Binding energy | | | | | |
|---------------------------------|----------------------|----------------------|----------------------|----------------------|---------------------|--|
| Electrocatalysts | Pt | | Ir | | Atomic Pt: Ir ratio | |
| | Pt 4f _{7/2} | Pt 4f _{5/2} | Ir 4f _{7/2} | Ir 4f _{5/2} | - | |
| $Pt_{3}Ir/Ti_{0.7}W_{0.3}O_{2}$ | 71.09 | 74.41 | 61.30 | 64.0 | 3.1:1 | |
| Pt ₃ Ir/C | 71.34 | 74.61 | 61.40 | 64.30 | 2.9: 1 | |
| Pt/C (E-TEK) | 71.19 | 74.38 | _ | - | _ | |



Figure S10. SEM image of the as-obtained $Pt_3Ir/Ti_{0.7}W_{0.3}O_2$ electrocatalyst.



Figure S11. EDX spectrum of the as-obtained $Pt_3Ir/Ti_{0.7}W_{0.3}O_2$ electrocatalyst.



Figure S12. TEM images of (a) Pt_3Ir/C and (b) commercial Pt/C electrocatalysts.



Figure S13. (a-c) Initial and 10000-cycle ADT curves of the differential electrocatalysts in N_2 -saturated 0.5 M H₂SO₄ aqueous solution at a scan rate of 50 mV s⁻¹.

| Catalysts | $ECSA^{(a)}$ $(m^2 g_{Pt}^{-1})$ | Onset potential ^(b) (V) | Mass activity ^(b) (mA mg _{Pt} ⁻¹) | I _f /I _b value ^(b) | Refs |
|--|----------------------------------|---------------------------------------|---|--|-----------|
| Pt ₃ Ir/Ti _{0.7} W _{0.3} O ₂ | 80.59 | 0.30 V vs. RHE | 810.03 | 10.09 | This work |
| Pt ₃ Ir/C | 63.97 | 0.46 V vs. RHE | 330.51 | 1.09 | This work |
| Pt/C (E-TEK) | 70.09 | 0.60 V vs. RHE | 219.48 | 0.84 | This work |
| EEG/Ppy-Pt ₃ Ni | 44.71 | - | 518.00 | 0.90 | 16 |
| Pt ₆₂ Pb ₂₈ /C | 50.00 | 0.70 V vs. RHE | 660.00 | 0.80 | 17 |
| Pt-AuSnO _x | 44.10 | 0.24 V vs. NHE | 302.00 | _ | 18 |
| Pt ₁ Rh ₁ ANDs | 66.30 | 0.65 V vs. RHE | 236.00 | 1.66 | 19 |
| Pt ₃ Co@Pt/PC | 67.00 | 0.35 V vs. SCE | 790.00 | 1.58 | 20 |
| d-PtIr/C | 71.05 | 0.34 V vs. RHE | - | 1.55 | 21 |
| Pt ₆₂ Pd ₃₈ /C NTDs | 44.60 | 0.7 V vs. RHE | 660.00 | 1.33 | 22 |
| Pt ₇₃ Pd ₂₇ /C | 25.35 | 0.40 V vs. RHE | 482.10 | 0.78 | 23 |
| PtRu@FeP (1 : 1) | 71.00 | 0.34 V vs. SCE | 653.00 | 0.98 | 24 |
| PtNiCu/C | 98.50 | 0.52 V vs. RHE | 632.00 | 0.90 | 25 |
| Pt/C-Cu ₃ P50% | 28.55 | 0.17 V vs. SCE | 413.96 | 0.91 | 26 |
| Pt-NiO/C-2 | 64.90 | 0.35 V vs. SCE | 644.00 | 0.92 | 27 |
| Pt ₃ Ru/Ti _{0.7} W _{0.3} O ₂ | 82.12 | 0.035 vs. NHE | 274.59 | 1.46 | 28 |

Table S3. A comparison of EOR activity of Pt-based electrocatalysts.

(a) Calculation from CV curves in N_2 -saturated 0.5 M H_2SO_4 aqueous solution.

^(b)Calculation from CV curves in N_2 -saturated 0.5 M H_2SO_4 + 1 M C_2H_5OH aqueous solution.



Figure S14. CV curves of (a) Pt_3Ir/C and (b) commercial Pt/C (E-TEK) electrocatalysts before and after bubbling CO in 0.5 M $H_2SO_4 + 1.0$ M C_2H_5OH aqueous solution at a scan rate of 25 mV s⁻¹.



Figure S15. CV curves of the $Pt_3Ir/Ti_{0.7}W_{0.3}O_2$, Pt_3Ir/TiO_2 , and $Pt/Ti_{0.7}W_{0.3}O_2$ catalysts in N₂-saturated 0.5 M H₂SO₄ + 1.0 M C₂H₅OH aqueous solution at a scan rate of 25 mV s⁻¹.



Figure S16. Electrocatalysis of the $Pt_3Ir/Ti_{0.7}W_{0.3}O_2$ and commercial PtRu/C for EOR (a) Mass activity and (b) Specific activity; (c) Comparison of the mass activity and specific activity at a scan rate of 25 mV s⁻¹ and (d) CA curves at fixed potential of 0.7 V vs. NHE for 10000 s in N₂-saturated 0.5 M H₂SO₄ + 1 M C₂H₅OH aqueous solution.



Figure S17. (a-c) 10000-cycle ADT at a scan rate of 50 mV s⁻¹ and (d) Normalized mass activity of the catalysts in N₂-saturated 0.5 M H₂SO₄ + 1 M C₂H₅OH aqueous solution.

Table S4. A comparison of catalytic stability of differential electrocatalysts after 5000 cycling test toward ethanol electrochemical oxidation.

| Catalysts | Mass activity (mA mg_{Pt}^{-1}) ^(a) | | $I_{\rm f}/I_{\rm b}$ value ^(a) | | Deterioration of | |
|---------------------------------|---|-----------------------------|--|-----------------------------|----------------------|--|
| | Before 10000 cycling test | After 10000 cycling test | Before 10000 cycling test | After 10000 cycling test | mass activity (%) | |
| $Pt_{3}Ir/Ti_{0.7}W_{0.3}O_{2}$ | 810.03 | 719.18 | 10.09 | 10.01 | 11.21 | |
| Pt ₃ Ir/C | 330.51 | 222.52 | 1.09 | 0.87 | 35.88 | |
| Pt/C (E-TEK) | 219.48 | 110.55 | 0.84 | 0.63 | 49.63 | |

^(a)Calculation from CV curves in N_2 -saturated 0.5 M $H_2SO_4 + 1$ M C_2H_5OH aqueous solution before and after 10000 cycling test.

| | Onset | Half-wave | Mass activity | Specific activity | |
|---|--------------------------|--------------------------|--|--|-----------|
| Catalysts | potential ^(a) | potential ^(a) | at 0.9 V _{RHE} ^(a) | at 0.9 V _{RHE} ^(a) | Refs. |
| | (V vs. RHE) | (V vs. RHE) | $(mA mg_{Pt}^{-1})$ | (mA cm ⁻²) | |
| $Pt_{3}Ir/Ti_{0.7}W_{0.3}O_{2}$ | 0.99 | 0.94 | 802.45 | 0.99 | This work |
| Pt ₃ Ir/C | 0.97 | 0.91 | 333.92 | 0.52 | This work |
| Pt/C (E-TEK) | 0.94 | 0.88 | 111.11 | 0.16 | This work |
| Pt ₃ Co/DMC-F | _ | 0.93 | 830.00 | 1.44 | 29 |
| Pt/PtP ₂ @NPC | _ | 0.89 | 724.00 | 0.51 | 30 |
| PtCo/Zn ₁₁ Co | _ | 0.92 | 460.00 | 0.72 | 31 |
| Commercial Pt/C | _ | 0.86 | 63.00 | 0.08 | 31 |
| Pd ₁ Pt ₄ DNSs | 0.99 | 0.89 | 530.00 | 0.74 | 32 |
| Pt-Co Concave NCs/C | _ | _ | 260.00 | 2.34 | 33 |
| USCS Au _{38.4} @Au _{9.3} Pt _{52.3} -NP/C | 1.02 | 0.89 | 750.00 | 0.72 | 34 |
| Pt _x Y-E/C | _ | 0.89 | 483.00 | 0.59 | 35 |
| PtCu-8 | _ | _ | 380.00 | 1.38 | 36 |
| La-doped Pt/C-5 | _ | _ | 490.00 | 0.93 | 37 |

Table S5. A comparison of oxygen reduction reaction (ORR) activity of Pt-based catalysts.

^(a)Calculation form LSV curves in O_2 -saturated 0.5 M H_2SO_4 aqueous solution at scan rate of 10 mV s⁻¹ with a rotating rate of 1600 rpm.

Before ADT test

After ADT test



Figure S18. TEM images of all electrocatalysts before and after 10000-cycle ADT in O_2 -saturated 0.5 M H_2SO_4 aqueous solution at a scan rate of 50 mV s⁻¹.

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