

**Electronic Supplementary Information for**

**Co-presence of PtNi Nanowires and Ionic Liquid in Carbon Mesopores  
Enhances Electrocatalytic Oxygen Reduction Activity**

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## Experimental section

**Materials.** Ethanol, cyclohexane, 2-propanol, perchloric acid, oleylamine, D(+)-glucose, 5% Nafion DE520 and nitric acid (13.0 M) were purchased from Wako Pure Chemical Industries, Ltd. [Pt(acac)<sub>2</sub>] and Pt/C (TEC10V30E) was purchased from Tanaka Kikinzoku Kogyo. Hexadecyltrimethylammonium chloride (CTAC) and [Mo(CO)<sub>6</sub>] were commercially available from TCI and Kanto Chemical, respectively. [Ni(acac)<sub>2</sub>], 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIM][PF<sub>6</sub>]), bis(trifluoromethane)sulfonimide lithium salt (Li[NTF<sub>2</sub>]) and 7-methyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene (MTBD) were purchased from Sigma-Aldrich. Pure oxygen gas (99.995%, Air Liquide Japan), pure argon gas (99.9995%, Hokkaido Air Water) and CO gas (99.9%, GL Science) were used for electrochemical measurements. Mesoporous carbon (abbreviated as mesoC) of CNovel MJ(4)010-00 (0.25 g) was purchased from TOYO TANSO. Vulcan XC-72R (abbreviated as C) was available from Cabot.

**Preparation of PtNi nanowires.** PtNi nanowires were prepared in a modified synthetic procedure based on reported synthetic procedures.<sup>1</sup> In 5 mL of oleylamine (15 mmol) in a two-necked round bottom flask (100 mL), [Pt(acac)<sub>2</sub>] (10 mg, 0.025 mmol), [Ni(acac)<sub>2</sub>] (6.5 mg, 0.025 mmol), CTAC (10 mg, 0.031 mmol), [Mo(CO)<sub>6</sub>] (20 mg, 0.075 mmol), and D-glucose (60 mg, 0.33 mmol) were added. The dispersion was kept at ca. 298 K in water under ultrasonication for 30 min and then stirred at 343 K for 2 min under the air. The reaction mixture was transferred to a pre-heated oil bath at 433 K and then kept with stirring at 433 K under the air for 2 h. The reaction mixture was naturally cooled down in the air to room temperature. A solution mixture (ca. 15 mL) of ethanol and cyclohexane (8:2, v/v) was added to the reaction mixture and then the mixture was centrifuged at 9,500 rpm at 298 K for 5 min using a Micro Refrigerated Centrifuge 3700 equipped with an angle rotor AF-5004CA (Kubota Co.). The supernatant was removed, and the isolated product was rinsed with ca. 15 mL of ethanol–cyclohexane solvent mixture (8:2, v/v) for at least five times under centrifugation at 9,500 rpm at 298 K for 5 min to obtain PtNi nanowires. The nanowires were stored in 5 mL of ethanol at room temperature until use. Yield: about 3 mg.

**Preparation of PtNi nanowires supported on carbon.** Vulcan XC-72R (2.3 mg) was dispersed in 15 mL of an ethanol-cyclohexane mixture (1:1, v/v) in an ice–water mixture under ultrasonication for 30 min. To the dispersion containing Vulcan XC-72R, a

dispersion containing 1 mg PtNi nanowires in ethanol was added and then the mixture was sonicated in an ice–water mixture for 4 h. The composite was centrifuged down at 9500 rpm at 298 K for 5 min and then rinsed with 15 mL of ethanol–cyclohexane solvent mixture (8:2, v/v) for at least five times under centrifugation at 9500 rpm at 298 K for 5 min to obtain Pt-Ni nanowires supported on Vulcan XC-72R (PtNi NW/C). PtNi NW/C was stored in 2 mL of ethanol at room temperature. For the preparation of catalyst ink, NW/C was dried at 393 K just before use.

PtNi NW/mesoC was prepared in the same procedure except for the use of mesoC. Before use of mesoC, 0.5 g of mesoC was treated with ball milling with 36.4 g of zirconia milling balls (0.1 mm in diameter) and ethanol (19 mL) at 450 rpm for 30 min 6 times using a planetary mill (PULVERISETTE 6, FRITSCH) and then dried under vacuum overnight.

**Preparation of [MTBD][NTf<sub>2</sub>].** In a 50 mL glass vial, MTBD (0.52 g, 3.39 mmol) was mixed with 10 mL of Milli-Q water. The solution was placed in an ice bath and nitric acid (13.0 M) was drop-wised at 273 K until the solution pH became 6–7. The solution was transferred into a 50 mL two-necked flask and then Li[NTf<sub>2</sub>] (0.975 g, 3.39 mmol) in 10 mL of Milli-Q water was added into the solution. The reaction mixture was stirred at 500 rpm under air overnight. The reaction mixture was rinsed with Milli-Q water using a separating funnel three times. The product was dried at 373 K under vacuum for at least 6 h, followed by drying at room temperature under vacuum overnight.

**Preparation of PtNi nanowires supported on carbon with ionic liquid.** An ionic liquid (IL) of [BMIM][PF<sub>6</sub>] (2.3 mg) and a carbon support, Vulcan XC-72R or mesoC (2.3 mg), were mixed in 15 mL of an ethanol-cyclohexane mixture (1:1, v/v) in an ice–water mixture under ultrasonication for 1 h. To the dispersion, a dispersion containing 1 mg PtNi nanowires in ethanol was added and then the mixture was sonicated in an ice–water mixture for 4 h. After the addition of about 15 mL of an ethanol-cyclohexane mixture (8:2, v/v) to the dispersion, IL/PtNi NW/C or IL/PtNi NW/mesoC was centrifuged down at 9,500 rpm at 298 K for 5 min using a Micro Refrigerated Centrifuge 3700 equipped with an angle rotor AF-5004CA (Kubota Co.). After repeating dispersion in the ethanol-cyclohexane mixture and centrifugation five times, and then the product was dried under vacuum overnight to obtain IL/PtNi NW/C or IL/PtNi NW/mesoC with a weight ratio of ionic liquid to carbon of 1 (IL/C = 1).

**Electrochemical measurements.** All electrochemical measurements were performed

using a conventional three-electrode setup. A potentiostat (SP-240, Biologic) was used to perform cyclic voltammetry and linear sweep voltammetry. A rotating disk electrode (RDE) was used as the working electrode. Platinum foil was used as the counter electrode. A Ag|AgCl (sat. KCl) electrode with a double junction holder (International chemistry Co., Ltd) or reversible hydrogen electrode (RHE) was used as the reference electrode. Catalyst-modified glassy carbon (GC) was used as the working electrode.

For preparation of working electrode, a head part of a GC screw (5 mm  $\phi$ , Tokai Carbon Co., Ltd.) was sonicated in ethanol for 5 min, polished with alumina slurry (0.3  $\mu\text{m}$ , Baikalex), followed by alumina slurry (0.05  $\mu\text{m}$ , Baikalex), and then sonicated in Milli-Q water for 5 min before drop-casting a catalytic ink. To prepare the catalyst ink with an ionomer/carbon ratio of 0.5 ( $I/C = 0.5$ ), 1.4 mg of the catalyst was dispersed in a mixture of 0.9 mL of 2-propanol, 1.6 mL of Milli-Q water, and 5% Nafion DE520 (10  $\mu\text{L}$ ) in a 50 mL glass vial in an ice-water mixture under ultrasonication for at least 2 h. The amount of the ionomer of Nafion was adjusted to the target  $I/C$  ( $I/C = 0, 0.25, 0.5$  or 1). The catalyst ink (20  $\mu\text{L}$ ) was drop-cast onto the GC screw that was rotating at 700 rpm using an AFMSRX Analytical Rotator and a MSRX Speed Controller (PINE Research Instrumentation), dried under rotating in a hot-air stream of a hair dryer for 3 min, and then kept at room temperature for 10 min (a metal loading density: about 17  $\mu\text{g cm}^{-2}$ ). The catalyst-modified GC screw was dried in the air at 418 K for 5 min in a furnace (FP22, Yamato Scientific) and then naturally cooled to room temperature before use for electrochemical measurements.

Cyclic voltammograms (CVs) and linear sweep voltammograms (LSVs) were recorded in an aqueous solution containing 0.1 M  $\text{HClO}_4$ . The electrolyte solution was purged with argon or oxygen for at least 30 min before electrochemical measurements. For electrochemical cleaning, 100 potential cycles at 200  $\text{mV s}^{-1}$  in the potential range between 0.05 and 1.20 V vs. RHE were performed in a 0.1 M  $\text{HClO}_4$  aqueous solution under Ar before collecting CVs and LSVs shown in the main text. All LSVs under oxygen are corrected by the subtraction of the current densities of the corresponding working electrode recorded under Ar. All potentials vs. Ag|AgCl (sat. KCl) were converted to potentials vs. RHE using the following equation:  $E$  (V vs. RHE) =  $E$  (V vs. Ag|AgCl (sat. KCl)) + 0.199 + 0.059·pH. For  $iR$  correction, electrochemical impedance spectroscopy (EIS) was performed at 0.5 V vs. RHE with frequencies ranging from 20 kHz to 1 Hz and an amplitude of 10 mV and 8 points/decade. A solvent resistance of 30~40  $\Omega$  was



determined using a software Z-Fit.

For CO stripping measurements, a potentiostat of HZ-7000 (Hokuto Denko) was used. Before CO adsorption, a 0.1 M HClO<sub>4</sub> aqueous electrolyte solution was purged with Ar for 30 min, the electrochemical cleaning of the catalyst was done as mentioned above, and then CVs were recorded under Ar. After that, the electrolyte solution was purged at with CO (5 sccm) for 10 min and then with Ar (120 sccm) for 60 min to remove CO in the electrolyte solution. A bias potential of 0.05 V vs. RHE was applied to a sample electrode during purging with CO and Ar until the measurements began. Cyclic voltammograms were recorded in the potential range from 0.05 to 1.45 V vs. RHE for two cycles at 50 mV s<sup>-1</sup> under Ar. Electrochemically active surface areas (ECSAs) based on charges of the electrochemical oxidation of CO (420 μC cm<sup>-2</sup>) and the desorption of the underpotentially deposited hydrogen (H<sub>upd</sub>) (210 μC cm<sup>-2</sup>) were determined from the difference between the first and second cycles.

**Physicochemical measurements.** Inductively coupled plasma mass spectrometry (ICP–MS) was performed using an ICP–MS spectrometer 8800 ICP–QQQ (Agilent Technologies). For sample preparation, a catalyst ink was dispersed in aqua regia solution and then the dispersion was stirred at 308 K for at least 24 h. The dispersion was filtered through a membrane filter (13HP045AN, ADVANTEC), and then the filtrate was diluted with Milli-Q water.

X-ray diffraction patterns were recorded at a voltage of 40 kV and a current of 40 mA using Cu Kα (λ = 0.154 nm) on RINT2000 (Rigaku). A sweep rate and a step width were 2° min<sup>-1</sup> and 0.01 °, respectively

X-ray photoelectron spectroscopy (XPS) data were collected at the pass energy of 10 eV using an Al Kα X-ray source by a photoelectron spectrometer JPS-9200 (JEOL). The binding energy was calibrated using C 1s at 284.6 eV. For the sample preparation, the catalyst ink was dropcast and then dried on the glassy carbon electrode. Before or after electrochemical measurements, the catalyst film was transferred onto the carbon tape and dried under vacuum.

STEM images were taken at an acceleration voltage of 200 kV using HD-2000 (Hitachi Scientific Instrument).

High angle annular dark-field scanning transmission–scanning transmission electron microscopy (HAADF–STEM) images and 3D electron transmission tomography images were taken using JEOL ARM-200F instrument which has ‘HR’-type pole pieces and

Cold-FE electron gun operated at 200 kV. The convergence angle was 34 mrad and the collection semi-angle was from 68 to 280 mrad. The electron probe current was about 120 pA. Tilt-series HAADF-STEM images for 3D tomography were taken from -60 to +46 degrees in step of 2 degrees, and a total of 53 images were acquired. The pixel size of the STEM images was 1024\*1024 pixels and the pixel length was 0.488 nm, thus the dimension of images was 499.7\*499.7 nm square. The duration time of electron beam was 25  $\mu$ s/pixel. We used the TEMography software package (SYSTEM IN FRONTIER INC.) for 3D STEM tomography. “STEM Recorder”, “Composer” and “Visualizer-evo” software were used for recording of tilt-series images, reconstruction of 3D volume data and rendering of 3D volume data, respectively. In the construction process of 3D volume data, tilt-series HAADF-STEM images were aligned each x-y positions using cross correction method, then calculated by iterative series reduction (ISER) method.

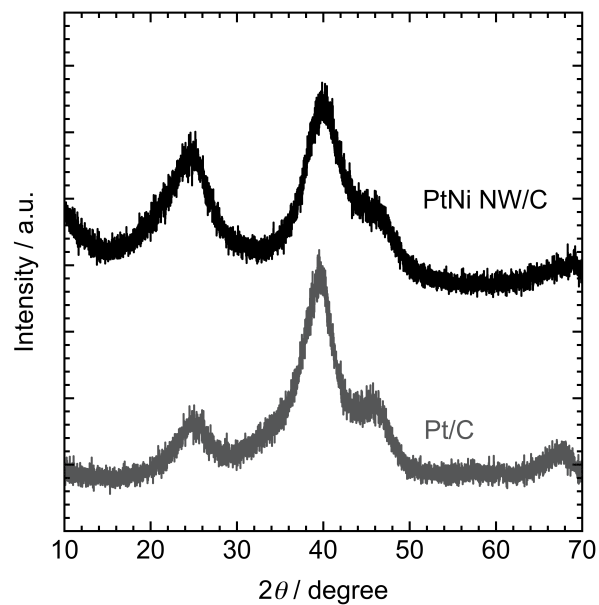
**Surface-enhanced infrared absorption (SEIRA) spectroscopy measurements.** SEIRA spectra were recorded on a Varian 7000 FT-IR spectrometer equipped with a HgCdTe detector (MCT), which was cooled with liquid nitrogen during the measurements. A polarization controller (SP01, Sankei Technics) and an infrared polarizer (GS12000, SPECAC) were used in front of the sample to obtain *s*- and *p*-polarized light beams,<sup>2</sup> where *s*- and *p*-polarized spectra were recorded and the *s*-polarized spectrum was used as the background to minimize signals originating from the bulk solution and maximize the signal intensity of the electrode surface. The polarization-modulated absorbance is defined as  $A = -\log(I_p/I_s)$ , where  $I_p$  and  $I_s$  are the single-beam intensities of the reflected *p*- and *s*-polarized infrared radiation, respectively. Differential spectra were obtained as  $A_{\text{sample}} - A_{\text{ref}}$ , where  $A_{\text{sample}}$  and  $A_{\text{ref}}$  are polarization-modulated absorbances for the sample and reference, respectively.

A Au/Si prism was prepared based on the procedure reported before.<sup>3,4</sup> For electrochemical cleaning, the Au/Si prism was attached to a home-made electrochemical glass cell, which can be placed in the ATR Kretschmann configuration in the spectrometer. A 0.05 M H<sub>2</sub>SO<sub>4</sub> aqueous solution was purged with Ar for 30 min and then 20 cycles of CVs of the Au/Si prism were repeated ranging between -0.4 and +0.95 V vs. RHE at a sweep rate of 20 mV s<sup>-1</sup> at 298 K, where the Pt counter and the Hg|HgSO<sub>4</sub> (sat. K<sub>2</sub>SO<sub>4</sub>) reference electrodes were used. The potentials vs. Hg|HgSO<sub>4</sub> (sat. K<sub>2</sub>SO<sub>4</sub>) were converted to those vs. RHE using the following equation:  $E$  (V vs. RHE) =  $E$  (V vs. Hg|HgSO<sub>4</sub> (sat.

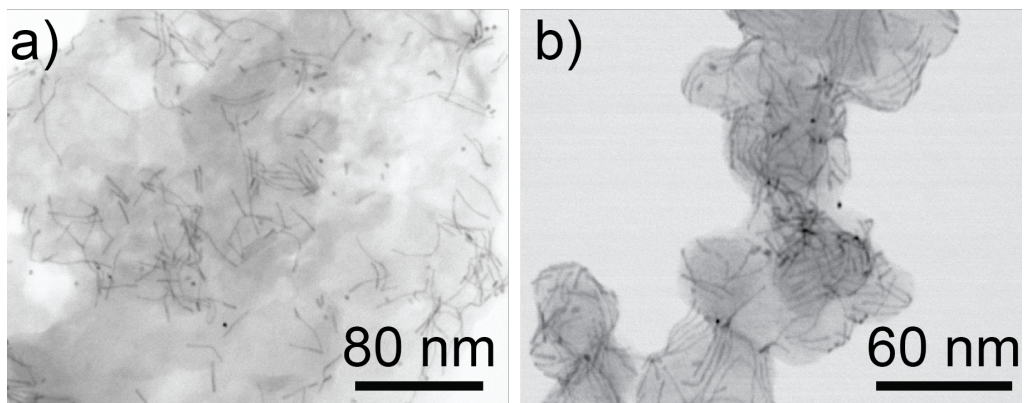
$\text{K}_2\text{SO}_4$ )) + 0.64 + 0.059 · pH.

After the electrochemical cleaning of the Au/Si prism, the electrolyte solution was gradually replaced by Milli-Q water. The Au/Si prism was rinsed with Milli-Q water and then dried on a hot plate at 343 K for 30 min to obtain 100  $\mu\text{L}$  of the catalyst ink. The prepared catalyst ink (20  $\mu\text{L}$ ) was diluted with the mixture of Milli-Q water (51.2  $\mu\text{L}$ ) and 2-propanol (28.8  $\mu\text{L}$ ) and then sonicated for 1 h in an ice-water mixture. After the Au/Si prism was heated at 373 K for 5 min, the diluted catalyst ink (100  $\mu\text{L}$ ) was drop-cast onto the preheated Au/Si prism at 373 K and then the prism was kept at 373 K for 3 min to dry the ink. The catalyst-modified Au/Si prism was heated in the furnace at 418 K for 5 min and then naturally cooled down outside the furnace to room temperature.

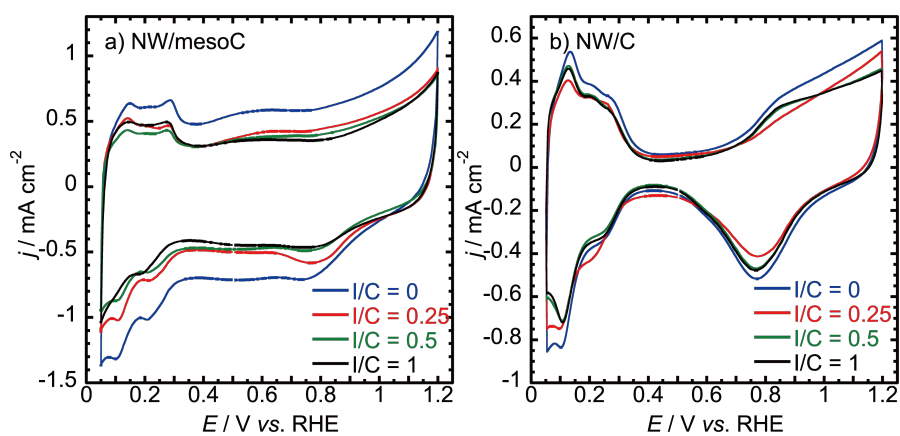
The catalyst-modified Au/Si prism was attached to the home-made electrochemical glass cell and was placed in the spectrometer. The electrochemical cell was filled with a 0.1 M  $\text{HClO}_4$  aqueous solution and the solution was purged with Ar for at least 30 min. After the electrochemical cleaning, potential-dependent SEIRA spectra of the catalyst-modified Au/Si prism were recorded. Each spectrum consists of 1024 interferograms, collected with a spectral resolution of 4  $\text{cm}^{-1}$ . Bias potentials were stepped ranging from 0 to 1.0 V vs. RHE at an interval of 0.1 V. After the spectra were recorded under Ar, the electrolyte was purged with CO at 0.05 V vs. RHE, and then with Ar for 1 h at 0.05 V vs. RHE until recording the spectra of the CO-adsorbed catalyst. The differential spectra at the same potential between the spectra before and after the CO adsorption to the catalyst were shown in this work.



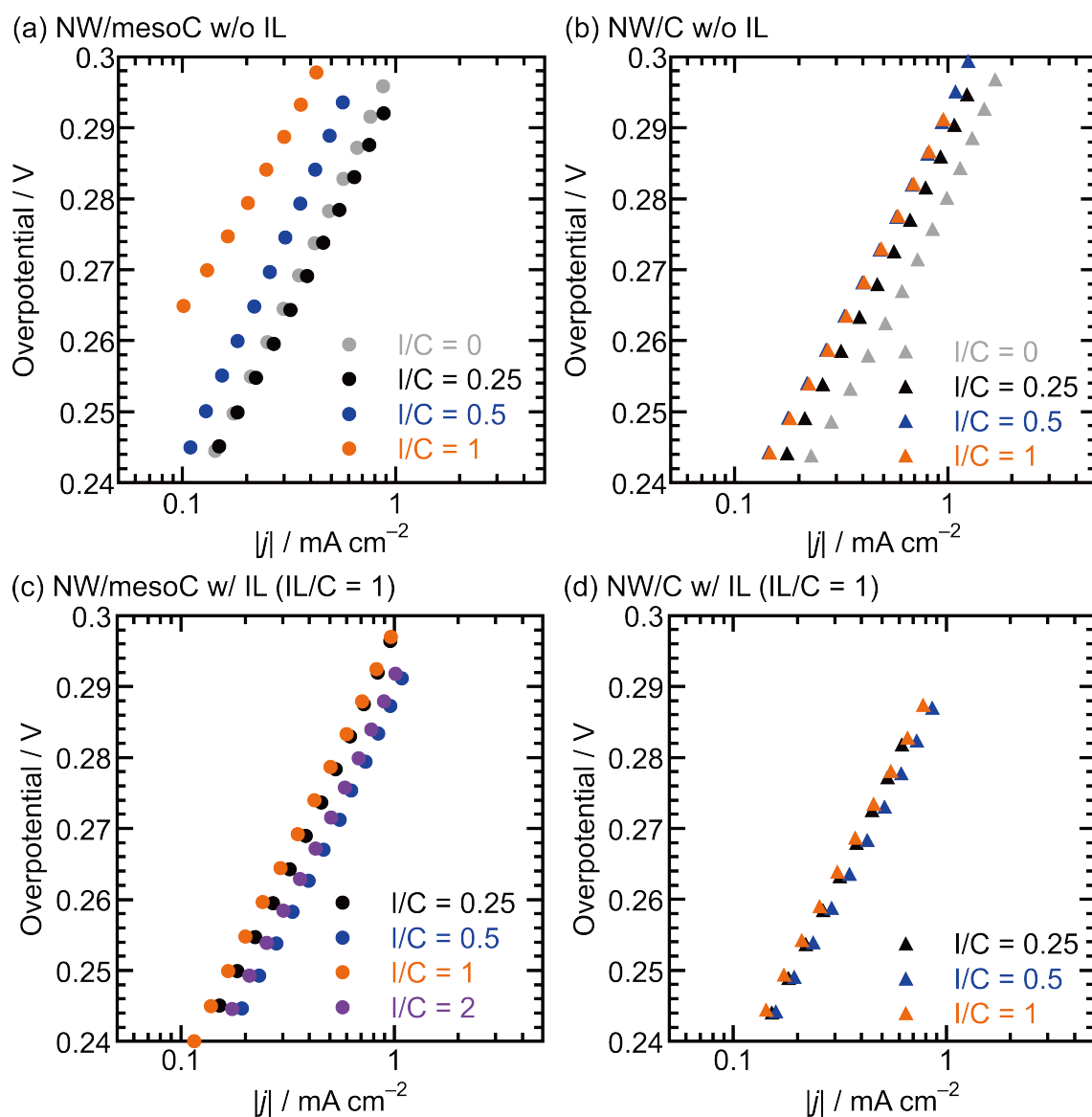
**Fig. S1** Powder XRD patterns of PtNi NW/C (the solid line in black) and Pt/C (TEC10V30E, TKK. The solid line in gray).



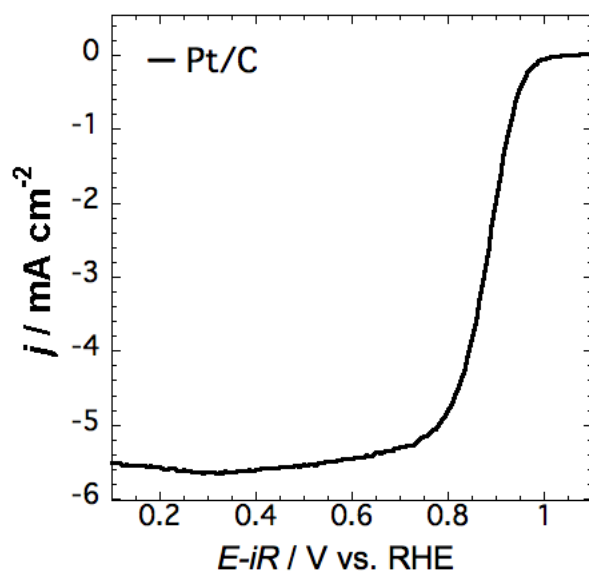
**Fig. S2** STEM images of (a) PtNi NW/mesoC and (b) PtNi NW/C.



**Fig. S3** CVs of (a) PtNi NW/mesoC and (b) PtNi NW/C for different I/C ratios with a scan rate of  $50 \text{ mV s}^{-1}$  in a  $0.1 \text{ M HClO}_4$  aqueous solution under Ar (IL/C=0).

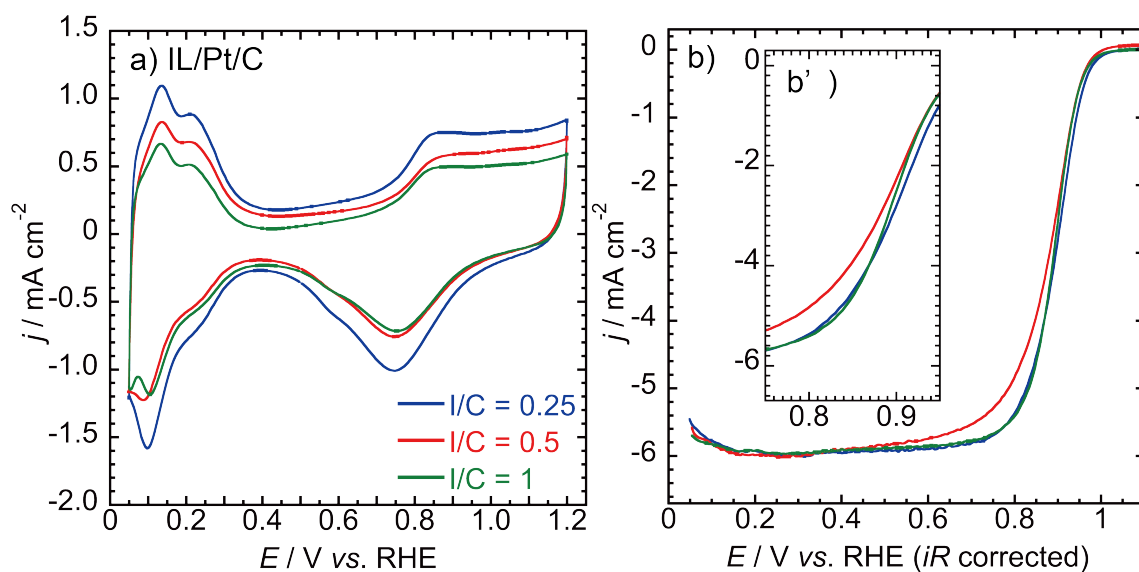


**Fig. S4** Tafel plots of (a) PtNi NW/mesoC (IL/C = 0), (b) PtNi NW/C (IL/C = 0), (c) PtNi NW/mesoC with IL (IL/C = 1) and (d) PtNi NW/C with IL (IL/C = 1). These plots are produced from LSVs from **Figs. 2** and **3** in the main text.

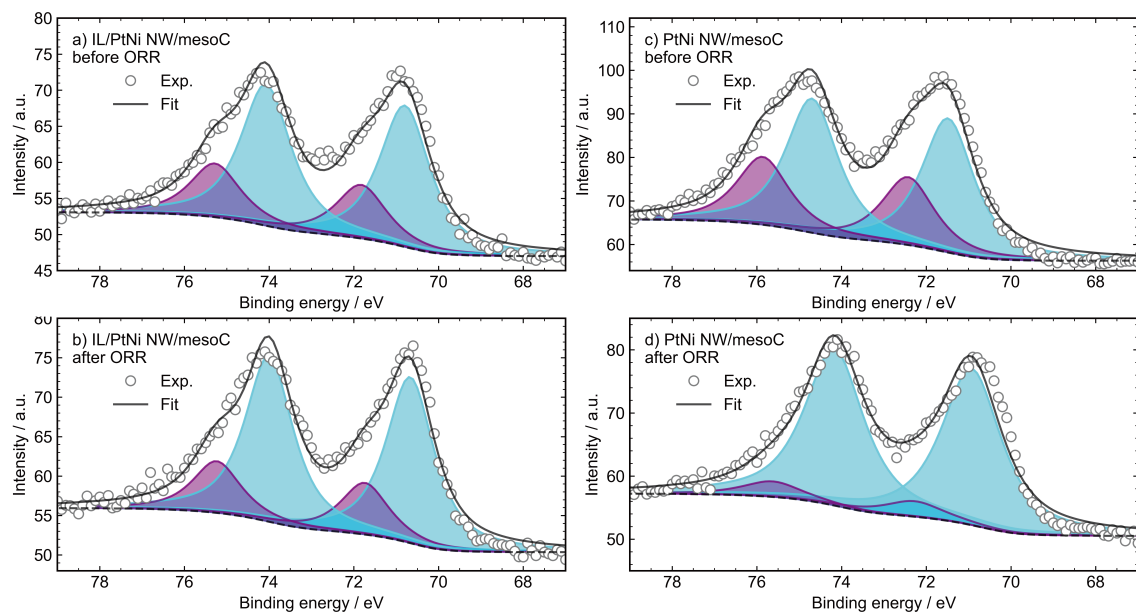


**Fig. S5** LSVs of commercially available Pt/C recorded at 1600 rpm and  $10 \text{ mV s}^{-1}$  in a  $0.1 \text{ M HClO}_4$  aqueous solution under  $\text{O}_2$  ( $I/C = 0.5$ ,  $IL/C = 0$ ).

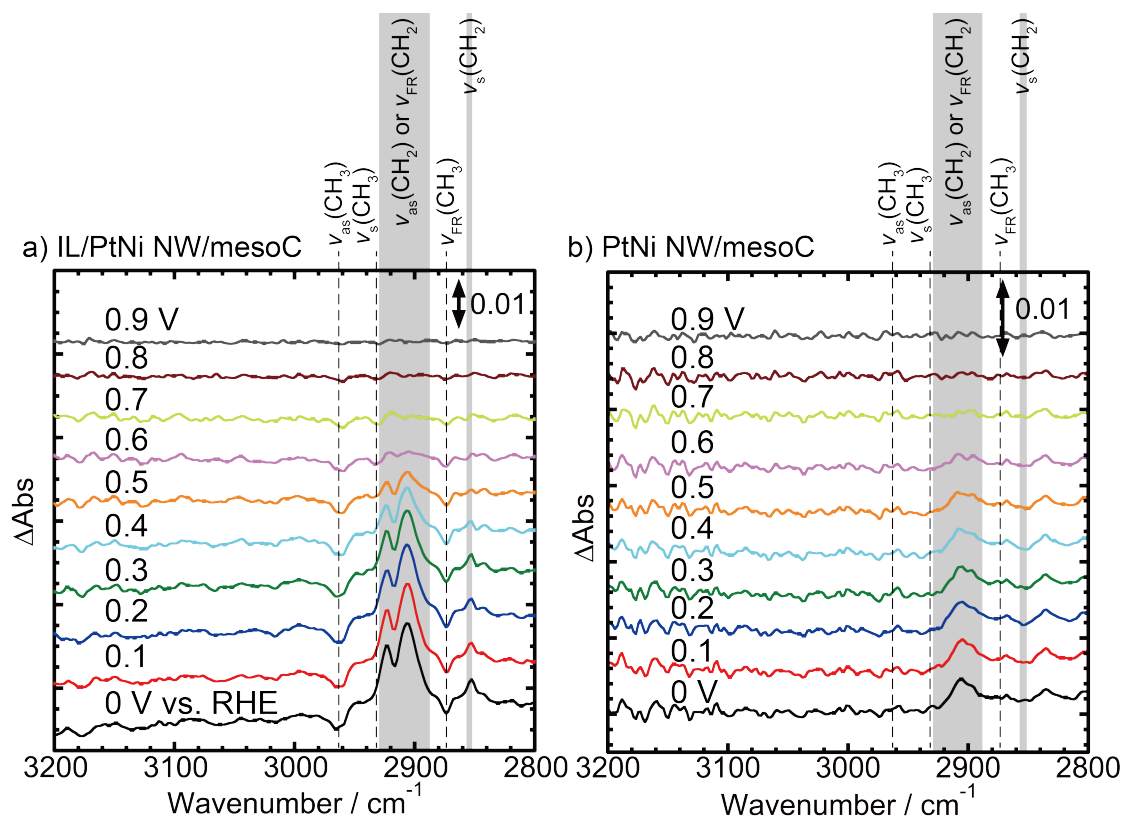




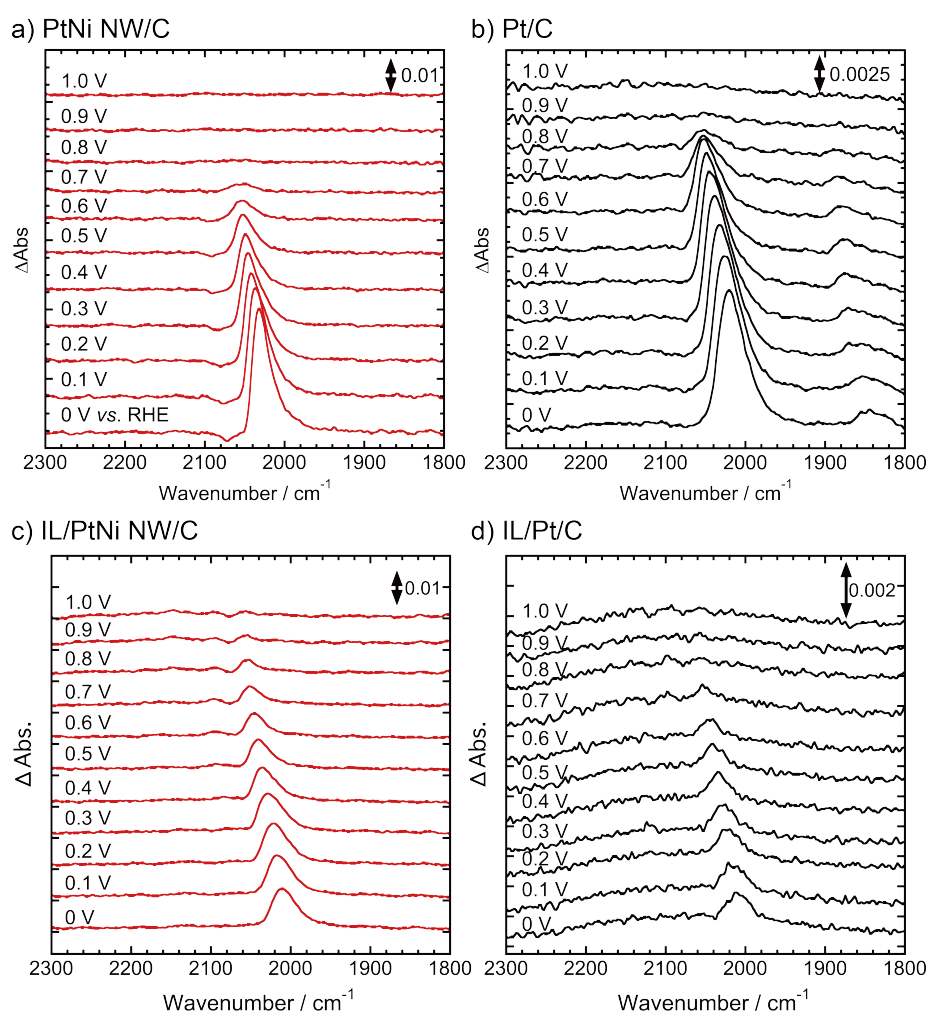
**Fig. S6** (a) CVs of IL/Pt/C with different I/C ratios (I/C=0.25 in blue, I/C = 0.5 in red and I/C = 1 in green) recorded at 50 mV s<sup>-1</sup> in a 0.1 M HClO<sub>4</sub> aqueous solution under Ar (IL/C = 1). (b) LSVs of IL/Pt/C with different I/C ratios (I/C=0.25 in blue, I/C = 0.5 in red and I/C = 1 in green) recorded at 1600 rpm and 10 mV s<sup>-1</sup> in a 0.1 M HClO<sub>4</sub> aqueous solution under O<sub>2</sub> (IL/C=1). The inset (b') show the enlarged LSVs. [BMIM][PF<sub>6</sub>] was used as the IL.



**Fig. S7** XPS data of IL/PtNi NW/mesoC ( $IL/C = 0.5$  and  $I/C = 0.5$ ) (a) before and (b) after ORR measurements and PtNi NW/mesoC ( $IL/C = 0$  and  $I/C = 0.5$ ) (c) before and (d) after ORR measurements. The open circles, solid lines in black and broken lines in black indicate the experimental data, fit data and background, respectively.



**Fig. S8** Potential-dependent SEIRA spectra of (a) IL/PtNi NW/mesoC (IL/C = 1, I/C = 1) and (b) PtNi NW/mesoC (IL/C = 0, I/C = 0.5). The spectra recorded at 1.0 V vs. RHE were used as the reference spectra. [BMIM][PF<sub>6</sub>] was used as the IL. The negative bands are assigned to the CH<sub>3</sub> groups<sup>5</sup> as shown by the broken lines. The labels of  $\nu_s(\text{CH}_3)$ ,  $\nu_{as}(\text{CH}_3)$  and  $\nu_{FR}(\text{CH}_3)$  indicate the symmetric stretching, asymmetric stretching and Fermi resonance modes of the CH<sub>3</sub> groups, respectively. The positive bands are associated with the CH<sub>2</sub> groups<sup>5</sup> as shown in the rectangles in gray. The labels of  $\nu_s(\text{CH}_2)$ ,  $\nu_{as}(\text{CH}_2)$  and  $\nu_{FR}(\text{CH}_2)$  indicate the symmetric stretching, asymmetric stretching and Fermi resonance modes of the CH<sub>2</sub> groups, respectively.



**Fig. S9** Potential-dependent SEIRA spectra of CO-adsorbed (a) PtNi NW/C and (b) Pt/C with no ionic liquid. The spectra of the catalysts with no CO-modification at the corresponding potential were used as reference spectra. IL/C = 0 and I/C = 0.5 for (a) and (b). Potential-dependent SEIRA spectra of CO-adsorbed (c) IL/NW/C and (b) IL/Pt/C. IL/C = 1 and I/C = 1 for (c) and (d). [BMIM][PF<sub>6</sub>] was used as the IL.

**Table S1.** MAs, SAs, and ECSAs of PtNi NW/mesoC and PtNi NW/C with different I/C ratios (IL/C=0).

Sample	I/C	MA	SA <sub>updH</sub>	SA <sub>CO</sub>	ECSA <sub>updH</sub>	ECSA <sub>CO</sub>
		/ A (mg-Pt) <sup>-1</sup>	/ mA (cm <sub>Pt</sub> ) <sup>-2</sup>	/ mA (cm <sub>Pt</sub> ) <sup>-2</sup>	/ m <sup>2</sup> (g <sub>Pt</sub> ) <sup>-1</sup>	/ m <sup>2</sup> (g <sub>Pt</sub> ) <sup>-1</sup>
PtNi NW/mesoC	0.25	0.70	0.94	0.52	74	135
	0.5	0.40	0.72	0.28	56	147
	1	0.21	0.22	0.14	93	151
PtNi NW/C	0.25	0.34	0.62	0.71	55	48
	0.5	0.32	0.52	0.60	62	53
	1	0.30	0.63	0.73	49	42

**Table S2.** Summary of Tafel slopes and exchange current densities ( $j_0$ ) obtained from **Fig. S3**.

	<b>IL/C</b>	<b>I/C</b>	<b>Tafel slope / mV (dec)<sup>-1</sup></b>	<b><math>j_0</math> / A cm<sup>-2</sup></b>
<b>PtNi NW/mesoC</b>	<b>0</b>	<b>0</b>	62.1	1.54 x10 <sup>-8</sup>
		<b>0.25</b>	68.9	4.63 x10 <sup>-8</sup>
		<b>0.5</b>	71.5	4.28 x10 <sup>-8</sup>
		<b>1</b>	53.9	1.28x10 <sup>-9</sup>
	<b>1</b>	<b>0.25</b>	64.4	2.43 x10 <sup>-8</sup>
		<b>0.5</b>	60.8	1.85 x10 <sup>-8</sup>
		<b>1</b>	61.7	1.49 x10 <sup>-8</sup>
		<b>2</b>	61.9	2.02 x10 <sup>-8</sup>
<b>PtNi NW/C</b>	<b>0</b>	<b>0</b>	49.7	2.38 x10 <sup>-9</sup>
		<b>0.25</b>	61.6	2.04 x10 <sup>-8</sup>
		<b>0.5</b>	58.8	1.04 x10 <sup>-8</sup>
	<b>1</b>	<b>1</b>	59.7	1.23 x10 <sup>-8</sup>
		<b>0.25</b>	62.6	1.94 x10 <sup>-8</sup>
		<b>0.5</b>	59.3	1.24 x10 <sup>-8</sup>
		<b>1</b>	57.9	8.56 x10 <sup>-9</sup>

**Table S3.** MAs and SAs of [MTBD][NTf<sub>2</sub>]-modified PtNi NW/mesoC with different IL/C ratios.

Sample	I/C	IL/C ratio	MA / A (mg-Pt) <sup>-1</sup>	SA <sub>updH</sub> / mA (cm <sub>Pt</sub> ) <sup>-2</sup>
[MTBD][NTf <sub>2</sub> ]/ PtNi NW/mesoC	0.25	1	0.26	0.88
		4	0.36	1.2
	1	1	0.18	0.76
		4	0.19	0.73

**Table S4.** MAs SA, and ECSAs of IL/PtNi NW/mesoC, IL/PtNi NW/C, and IL/Pt/C with different I/C ratios (IL/C = 1), where [BMIM][PF<sub>6</sub>] was used as the IL. MAs and SAs were determined at 0.9 V vs. RHE (*iR* corrected).

Sample	I/C ratio	MA	SA <sub>upDH</sub>	SA <sub>CO</sub>	ECSA <sub>upDH</sub>	ECSA <sub>CO</sub>
		/ A (mg-Pt) <sup>-1</sup>	/mA (cm-Pt) <sup>-2</sup>	/ mA (cm-Pt) <sup>-2</sup>	/ m <sup>2</sup> (g-Pt) <sup>-1</sup>	/ m <sup>2</sup> (g-Pt) <sup>-1</sup>
IL/PtNi NW/mesoC	0.25	0.58	0.45	0.40	129	143
	0.5	0.56	0.88	0.48	64	117
	1	0.79	0.58	0.44	136	182
	2	0.87	1.4	0.60	64	145
IL/PtNi NW/C	0.25	0.19	0.43	0.73	45	27
	0.5	0.53	0.54	0.61	99	87
	1	0.23	0.60	0.68	38	33
IL/Pt/C	0.25	0.35	-	-	-	-
	0.5	0.27	-	-	-	-
	1	0.26	-	-	-	-



**Table S5.** Summary of fitting parameters for XPS peak deconvolution analysis, shown in **Fig. S7.**

<b>Region</b>	<b>Position / eV</b>	<b>Area / a.u.</b>	<b>Relative amount / %</b>	<b>Assignments</b>
<b>IL/PtNi NW/mesoC before ORR measurements</b>				
Pt 4f <sub>7/2</sub>	70.8	41.1	72	Pt(0)
	71.8	15.6	28	Pt(II)
Pt 4f <sub>5/2</sub>	74.1	41.1	72	Pt(0)
	75.3	15.6	28	Pt(II)
<b>IL/PtNi NW/mesoC after ORR measurements</b>				
Pt 4f <sub>7/2</sub>	70.7	41.9	76	Pt(0)
	71.7	13.1	24	Pt(II)
Pt 4f <sub>5/2</sub>	74.0	41.9	76	Pt(0)
	75.2	13.1	24	Pt(II)
<b>PtNi NW/mesoC before ORR measurements</b>				
Pt 4f <sub>7/2</sub>	71.5	67.1	66	Pt(0)
	72.4	34.0	34	Pt(II)
Pt 4f <sub>5/2</sub>	74.7	67.1	66	Pt(0)
	75.9	34.0	34	Pt(II)
<b>PtNi NW/mesoC after ORR measurements</b>				
Pt 4f <sub>7/2</sub>	70.9	61.4	91	Pt(0)
	72.3	6.1	9	Pt(II)
Pt 4f <sub>5/2</sub>	74.2	61.4	91	Pt(0)
	75.6	6.1	9	Pt(II)

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