Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information

Synthesis of Monolayer Platinum Nanosheets

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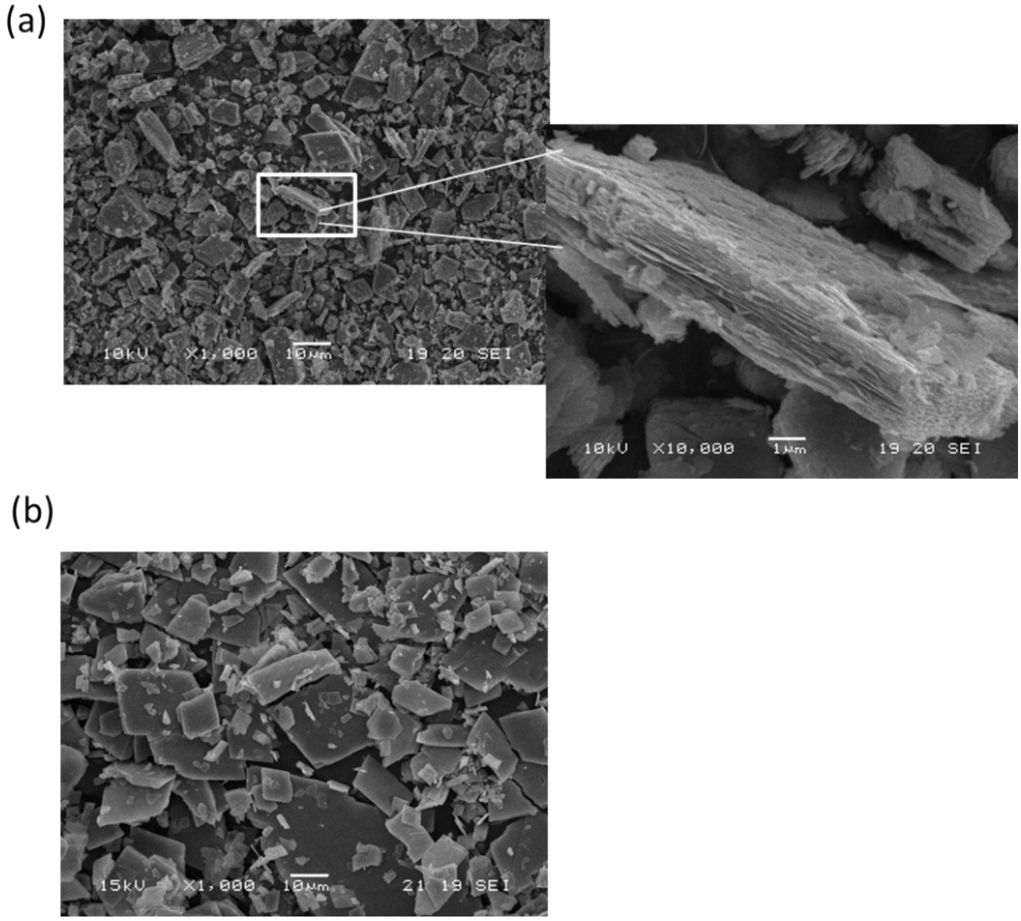

Fig. S-1 SEM image of layer compounds of in the presence (a) and absence (b) of SDS.

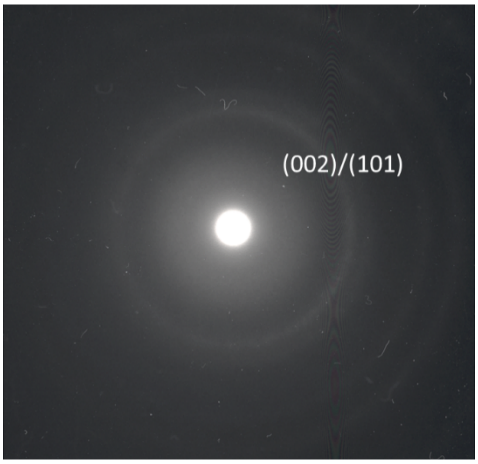

Fig. S-2 SAED patterns of layer compounds.S-1) and S-3)

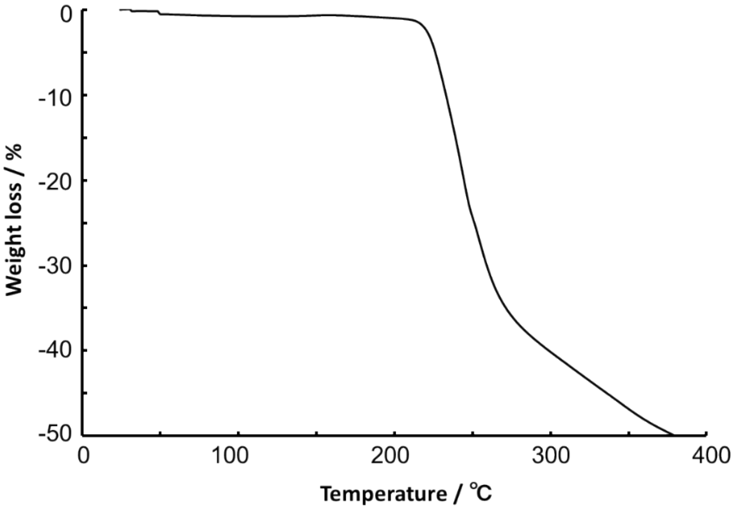

Fig. S-3 Thermal analysis (TG) results of the layered oxide. TG curve of layered Pt precursor intercalated with $(CH_2)_6H_xN_4^{x+}$ ions recorded in air atmosphere. The first stage of weight loss below 215 °C was 1.3 %, which is due to the removal of interlayer water molecules. The latter stage of weight loss is due to removal of interlayer $(CH_2)_6H_xN_4^{x+}$ ions.

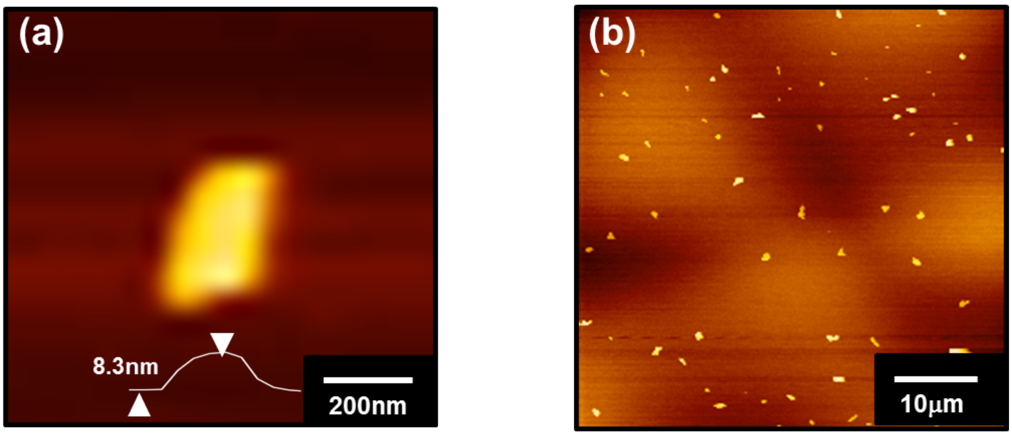

Fig. S-4 AFM image of non-DS nanosheets.

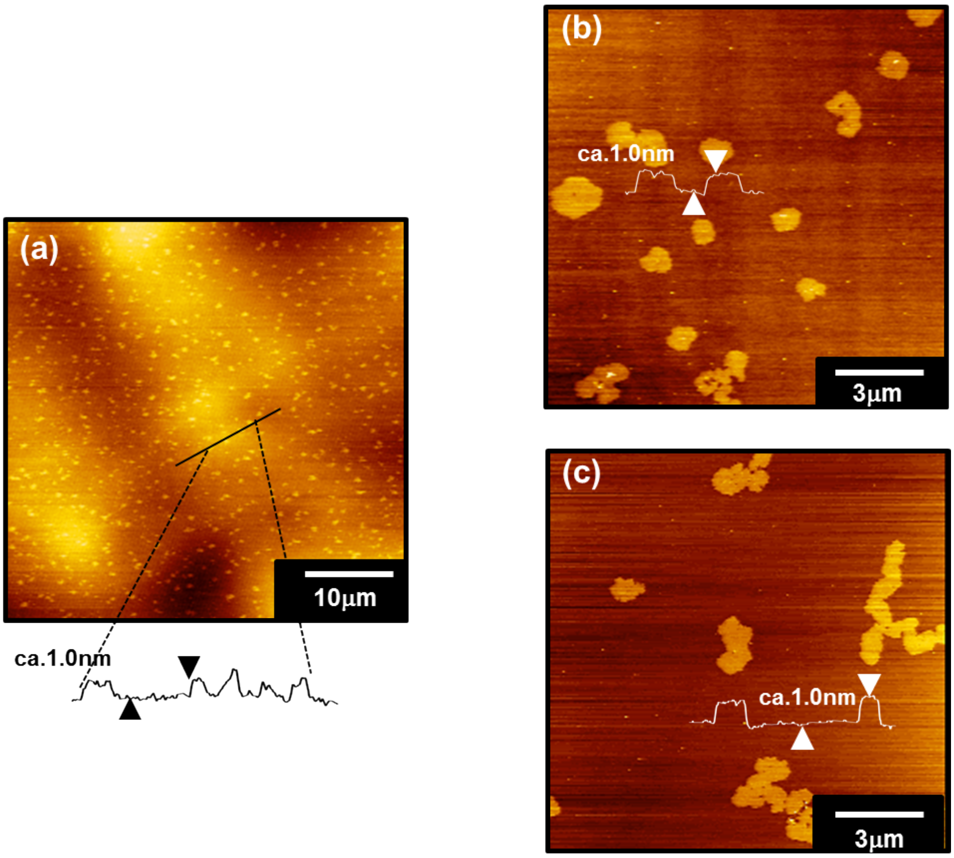

Fig. S-5 AFM images of DS-nanosheets after electrochemical reduction.

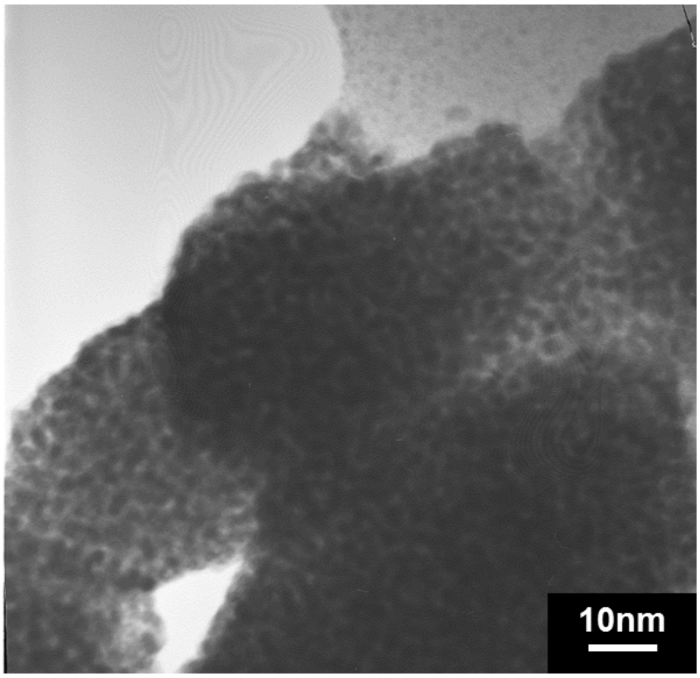

Fig. S-6 TEM image of DS-nanosheets after chemical reduction.

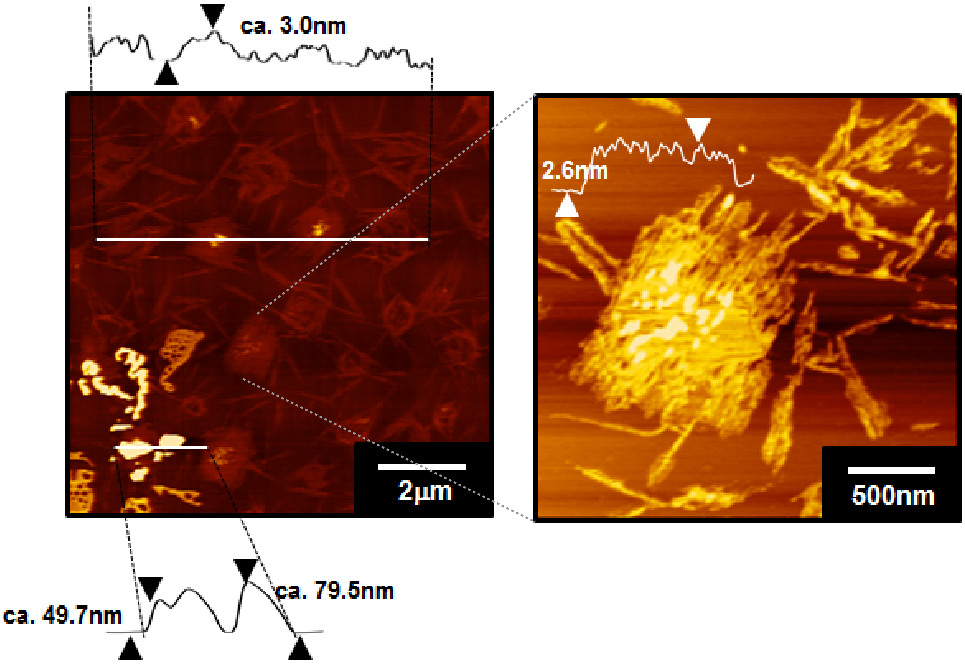

Fig. S-7 AFM images of DS-nanosheets after chemical reduction.

The thickness of Ds-nanosheets after chemical reduction were approximately from 2 nm to 80 nm.

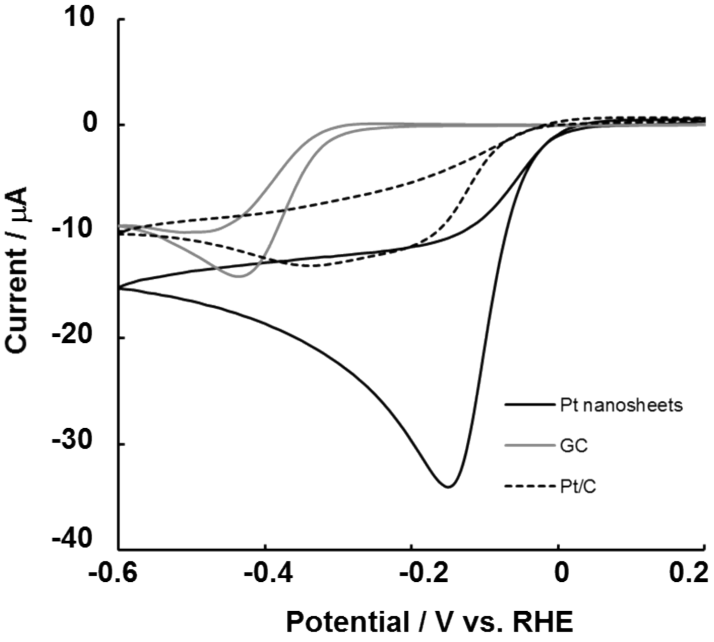

Fig. S-8 CV curves of the monolayer Pt-nanosheets (Pt:0.12μg), Pt/C(Pt:0.12μg), and glassy carbon electrode in O^{2-} saturated 0.1M KOH. .

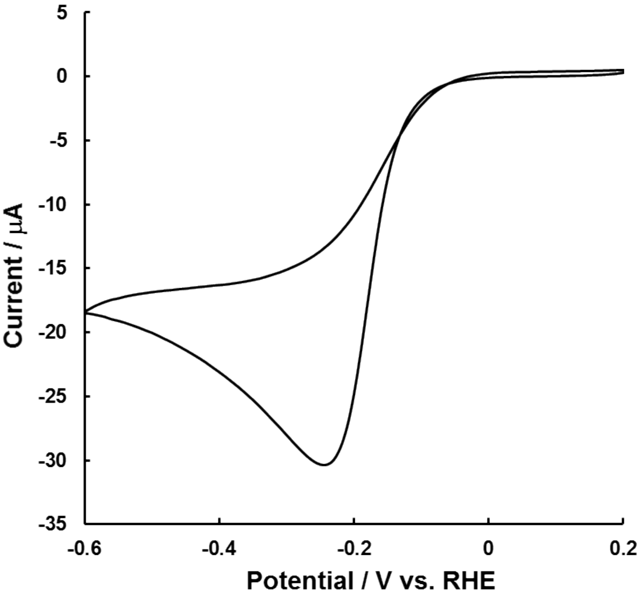

Fig. S-9 CV curves of the DS-nanosheets after chemical reduction on glassy carbon electrode in O^{2-} saturated 0.1M KOH. .

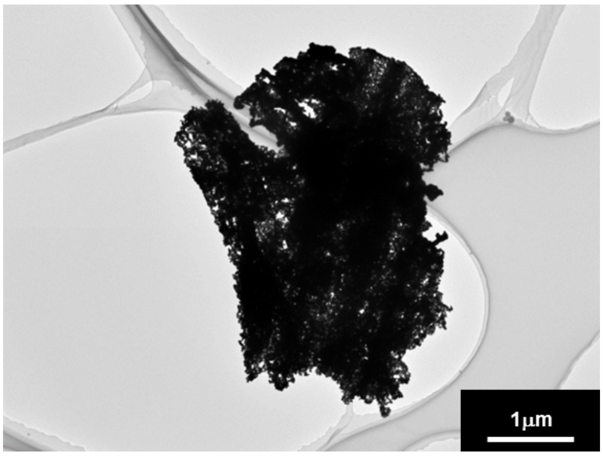

Fig. S-10 TEM image of DS-nanosheets after chemical reduction.

Table S-1. XPS spectra of layer compounds of in the presence and absence of SDS.

(a) Pt4f peak of layer compounds of in the presence and absence of SDS.

	Pt(O)	PT(+II)	Pt(+IV)	Pt(+II)
		PtO	PtO_2 or $PtCl_6^{4-}$	$PtCl_4^{2-}$
presence SDS	0	95.2	0	4.8
absence SDS	0	95.6	0.7	3.7

(b) Main peak spectra of layer compounds of in the presence and absence of SDS except Pt 4f.

	C	O	Pt	Cl	N	S	Na	B	K
presence SDS	57.3	2.9	3.7	8	27.6	0.5	-	-	0
absence SDS	54.8	2.1	4	8.6	30.3	0.2	-	-	0

Experimental.

The nanosheet precursors, i.e., the layered compounds, were prepared in the presence and absence of SDS. The two types of layered compounds were synthesized by mixing an aqueous solution of K_2PtCl_4 (0.05M), hexamethylenetetramine (HMT; 1M), and SDS (0.5M) as follows: Aqueous (deionized water) solutions of K_2PtCl_4 (12 ml), SDS (10 ml), and HMT (6 ml) were mixed in a 100 cm^3 vessels, and the mixtures were set aside for 24 h at room temperature. Subsequently, the mixtures were centrifuged (3000 rpm, 20 min), and the precipitates rinsed with distilled water and ethanol, and then dried at room temperature. Each precipitated layered compound was exfoliated in ethanol and tetrabutylammoniumhydroxide (TBAOH) aqueous solutions. The Pt nanosheet precursors were subsequently reduced either chemically ($NaBH_4$) or electrochemically (reduction condition; at −1.0 V (Ag/AgCl) in 0.1M Na_2SO_4, reduction time; 3600 s). Preparation of the Pt nanosheets/GC electrode, as a working electrode, was reduced for 5 min at −1.0 V (Ag/AgCl) in 0.1 M Na_2SO_4 with Pt wire as a counter electrode in an electrochemical reduction process. Preparation of the Standard Pt/C Electrode : The Pt/C catalyst (30 mg, 20 wt% Pt on Vulcan XC-72; ElectroChem, Inc.) was suspended in a solution of 10 wt% nafi on (0.12 mL) in ethanol (10 mL), and a droplet of the resulting suspension involving 75 μ g of catalyst was placed on a GC electrode surface and then vacuum-dried. Note that we carefully fabricated the reference electrode such that the ORR activity of the Pt/C catalyst employed here was closely comparable with that reported elsewhere measured under the same conditions.[S-3)] Electrochemical Analysis: Cyclic voltammetry (CV) measurements were carried out using three-electrode electrochemical cells. Samples were tested as electrodes for the oxygen reduction reaction (ORR) in 0.1 M KOH saturated with O_2. Pt wire and Ag/AgCl electrodes were used as the counter and reference electrodes, respectively.

The crystal structure was analyzed using XRD patterns (Rigaku RINT-2500VHF, equipped with a Cu Kα radiation source). The concentration of the nanosheets in the solution was analyzed by an inductively coupled plasma (ICP) spectrometer (Thermo Fisher Scientific, IRIS Advantage DUO). The thickness of the delaminated nanosheets was confirmed by AFM (Nanocute, SII NanoTechnology, tapping mode) measurements. The samples for the AFM observations were prepared by casting of the diluted nanosheet solution onto mica substrates. The samples were dried under pure N_2 at room temperature. For TEM (Hitachi, HF-2000, 200 kV) observations, one drop of the aqueous nanosheet suspension was deposited onto a holey carbon film. XPS (Thermo Scientific, Sigma Probe) was used to analyze the layered oxides and nanosheets. A monochromatized X-ray source (Al Kα, hν = 1486.6 eV) was used for the XPS measurements. For these measurements, an Au substrate (in nanosheet/Au film) was used to determine the Fermi level. The nanosheet concentration in the solution was analyzed by an inductively coupled plasma spectrometer (Seiko Instruments, SPS7800), in which the nanosheets were dissolved in concentrated HCl before measurement. UV–vis absorption spectra were obtained using a Jasco V-550 spectrometer. Thermogravimetric–differential thermal analysis curves for the layered oxides were obtained by thermal analysis (Seiko TG/DTA).

S-1) J. R. McBride, G. W. Graham, C. R. Peters, W. H. Weber, *J. Appl. Phys.* 1991, **69**, 1596-1604

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S-3) R. K. Nomiyama, M. J. Piotrowski, J. L. F. D. Silva, Phys. Rev. B 2011, **84**, 100101(R), 1-4

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