

# NEW PATENTS

## CATALYSIS – APPLIED AND PHYSICAL ASPECTS

### Preparation of Platinum on Activated Carbon

UNIV. KEBANGSAAN MALAYSIA *World Appl.* 2009/057,992

A catalyst with  $\geq 40$  wt.% loading of Pt and mean particle size of  $28 \mu\text{m}$  is prepared by adding a solution of  $\text{H}_2\text{PtCl}_6$  or  $(\text{NH}_4)_2\text{PtCl}_4$  in *aqua regia* to activated C powder with particles  $20\text{--}30 \mu\text{m}$  in size, pretreated with  $\text{HNO}_3$ . A base such as  $\text{NH}_4\text{OH}$  is added to raise the pH to  $9.7\text{--}9.9$ . The solution is boiled and calcined by heating at  $120\text{--}130^\circ\text{C}$  then at  $340\text{--}360^\circ\text{C}$ .

### Palladium-Gallium Hydrogenation Catalysts

MAX-PLANCK-GESELLSCHAFT *World Appl.* 2009/062,848

Optionally supported, ordered intermetallic PdGa compounds are prepared by reacting a Pd compound, preferably  $\text{Pd}(\text{acac})_2$ , with a Ga compound, preferably a Ga halide, in the presence of a reductant such as  $\text{LiBEt}_3\text{H}$  and optionally a solvent such as THF or diglyme. Alternatively, Pd is reacted with a vaporised Ga compound, such as  $\text{GaI}_3$ . Particular application for the selective conversion of ethyne to ethene in the presence of an excess of ethene is claimed.

## CATALYSIS – INDUSTRIAL PROCESS

### Two-Stage Distillate to Gasoline Conversion

CONOCOPHILLIPS CO *U.S. Appl.* 2009/0,134,061

Distillate with research octane number (RON)  $25\text{--}50$ , is converted to gasoline with  $\text{RON} > 65$ , by contact with: (a)  $0.5\text{--}5$  (preferably  $0.5\text{--}3$ ) wt.% Pt and/or Pd on an acidic support, preferably zeolite, in the presence of  $\text{H}_2$ , at  $220\text{--}260^\circ\text{C}$ , then (b)  $0.5\text{--}5$  (preferably  $0.5\text{--}3$ ) wt.% Ir and optionally Ni on a support such as  $\text{SiO}_2$  or  $\text{Al}_2\text{O}_3$ , at  $280\text{--}330^\circ\text{C}$ .

## CATALYSIS – REACTIONS

### Iridium Catalyst for Nitrile Hydration

UNIV. OKAYAMA *Japanese Appl.* 2009-023,925

Amides are produced from nitriles under mild conditions using a catalyst system formed from an Ir complex,  $\text{XIrL}_2$ ,  $\text{YIrZ}_2$  or  $(\text{YIrZ})_2$ , with an electron-withdrawing organic phosphine; where X = a Group 15 element or a bidentate ligand containing  $\text{O}^-$ ; L = a phosphine or a neutral ligand exchangeable with a phosphine; Y = a multidentate ligand containing  $\text{C}^-$  or  $\text{N}^-$ ; and Z = a negatively charged monodentate ligand.

## EMISSIONS CONTROL

### Efficient Treatment of Particulate Matter

ETM INT. LTD *World Appl.* 2009/090,447

A catalyst cartridge contains mineral fibres with density  $300\text{--}1000 \text{g m}^{-3}$ , composed of  $\geq 80$  wt.%  $\text{SiO}_2$  with Pt and/or Ir, arranged in radial undulations with rectangular sections of length  $l$  perpendicular to the direction of gas flow. The fibres are  $0.3\text{--}1 \text{mm}$  thick, with distance  $d$  between undulations such that  $l/d = 5\text{--}12$ .

### Lean NOx Trap and Reduction Catalyst

TOYOTA MOTOR CORP *Japanese Appl.* 2009-028,575

A  $\text{NO}_x$  occlusion and reduction catalyst has Rh supported on two different oxide materials, such as a mixture of  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2\text{-TiO}_2$ , in the ratio  $1.9\text{--}5:1$ . Rh solubility is  $\geq 70\%$  in the first and  $< 70\%$  in the second oxide, when heat treated at  $> 750^\circ\text{C}$  and at a loading of  $0.01\text{--}5$  wt.% Rh. During lean operation, Rh goes into solid solution in the first oxide, preventing grain growth and sintering. In the rich phase, Rh precipitates out of solution to catalyse  $\text{NO}_x$  reduction.

## FUEL CELLS

### Membrane Electrode Assembly Evaluation

GM GLOBAL TECHNOL. OPER. INC

*U.S. Appl.* 2009/0,124,020

A PEMFC MEA is soaked in an unsaturated organic compound, such as  $0.5\text{--}2.0$  wt.% polyoxyethylene (10) oleyl ether (Brij<sup>®</sup> 97) in  $\text{H}_2\text{O}$ , and then stained with a strongly oxidising agent, specifically  $\text{OsO}_4$ . The MEA is embedded in an epoxy and thin sections for viewing using TEM are prepared. Ionomer and catalyst particles will appear as dark regions and pores as light regions, allowing porosity and size and distribution of particles to be determined.

### Membrane Electrode Assembly with Anion Exchange

TOSHIBA CORP

*Japanese Appl.* 2009-026,690

A membrane/electrode composite includes an anion exchange substance, deposited on the anode catalyst layer or the electrolyte membrane, which captures mobile Ru-containing anions to prevent catalyst degradation. The substance is deposited as a film, or as particles with diameter  $0.01\text{--}50 \mu\text{m}$ , in mass ratio of  $5:95\text{--}90:10$  relative to the anode catalyst, or at a loading of  $1\text{--}50 \text{mg cm}^{-2}$  of electrode surface area.

## METALLURGY AND MATERIALS

### Platinum Jewellery Alloy

HEIMERLE & MEULE GmbH *World Appl.* 2009/059,736

A Pt alloy consists of (in wt.%):  $94.0\text{--}96.5$  Pt (preferably  $95.1\text{--}95.5$ );  $2.5\text{--}4.5$  W (preferably  $3.7\text{--}3.8$ );  $0.5\text{--}3.0$  Cu (preferably  $0.9\text{--}1.1$ ); and  $0.02\text{--}2.0$  (preferably  $0.04\text{--}0.07$ ) of at least one of Ru, Rh and Ir, and is free of Au. It offers high hardness and resistance to abrasion combined with good workability and cold formability. Semi-finished jewellery components are also claimed.

### Corrosion-Resistant Platinum-Rhodium Alloy

ISHIFUKU MET. IND. CO, LTD *Japanese Appl.* 2009-035,750

A PtRh alloy for high-temperature and electrical applications is described as possessing good resistance to corrosion caused by P, Pb, As, B, Bi, Si, Zn, but in particular P. It consists of  $10\text{--}40$  wt.% Rh with at least one of (in wt.%):  $0.1\text{--}5.0$  V, Cr, Nb, Mo, Ta, Re and/or W;  $0.1\text{--}3.0$  Mn and/or Co;  $0.3\text{--}5.0$  Ru, Pd, Ir, Au and/or Ag; and/or  $0.01\text{--}1.0$  Al, and the balance Pt.

## APPARATUS AND TECHNIQUE

### Spark Plug with Iridium Alloy Tip

HONEYWELL INT. INC *U.S. Appl.* 2009/0,127,996

A spark plug with electrode tip formed from an Ir alloy is presented. The alloy composition is (in wt.%): 60–70 Ir, 30–35 Rh, 0–10 Ni and has minor additions (in ppm) either: (a) 3500–4500 Ta and 100–200 Zr, or (b) 50–100 Ce. These allow for better bonding of the tip with the Ni alloy of the electrode body through interdiffusion. The Ir alloy offers high wear resistance.

### Unsupported Palladium Membranes

J. D. WAY *et al.* *U.S. Appl.* 2009/0,176,012

Defect-free, 7.2  $\mu\text{m}$ -thin Pd membranes are formed by electroless plating on a support such as mirror-finished stainless steel, in an EDTA-free plating bath at 50°C with addition of 1 part per 100 of hydrazine solution (3 M). The support may be seeded with metallic Pd crystallites from Pd acetate. A second metal such as Cu, Ag or Au may also be deposited by electroless plating and the membrane homogenised by annealing. The membrane is freed from the support by scoring the edges.  $\text{H}_2$  permeabilities may be equivalent to thicker membranes and  $\text{H}_2/\text{N}_2$  selectivity can reach 40,000.

### Porous Platinum Nanoparticles

UNIV. MIYAZAKI *Japanese Appl.* 2009-062,571

Monocrystalline Pt nanoparticles with nanopores, for use in catalysts, electrodes or sensors, and their method of production are described. The particles are sheets 2–25 nm thick with outer diameter 30–600 nm. The pores may have diameter 1–3.5 nm or may be elliptical or rectangular with dimensions  $1 \times 3.5$  to  $3.5 \times 10$  nm, and are arrayed at regular intervals of 4–5 nm or at intervals varying from 1–5 nm. The particles may be composed of Pt, Pt and a base metal, or an alloy of Pt with Pd, Rh, Ir, Ru, Au, and/or Ag.

## BIOMEDICAL AND DENTAL

### Anticancer Rh(III) and Ir(III) Complexes

FREIE UNIV. BERLIN *European Appl.* 2,072,521

Novel octahedral metal(III) polypyridyl complexes  $\text{M}(\text{hal})_3(\text{sol})(\text{pp})$  for the prevention and treatment of cancer and its metastases are claimed, described as exhibiting superior cytotoxic activity in cell cultures. M is Rh or Ir, preferably Rh. Sol is a solvent, preferably DMSO or  $\text{H}_2\text{O}$ ; hal is a halogen, preferably Cl or Br, or a pseudohalogenide, preferably SCN; and pp is a polypyridyl ligand, preferably dpq, dppz or dppn.

### Ultra-Low Magnetic Susceptibility Palladium Alloys

DERINGER-NEY INC *U.S. Appl.* 2009/0,191,087

Pd alloys for biomedical components compatible with the use of magnetic resonance imaging are claimed. The composition is (at.%):  $\geq 75$  Pd; 3–20 Sn, Al or Ta; plus one or more of whichever of Sn, Al or Ta are not used in the binary composition, and/or Nb, W, Mo, Zr or Ti, up to a total of 22 at.%. The alloys are formulated such that the volume magnetic susceptibility (cgs) is between  $3 \times 10^{-6}$  and  $-3 \times 10^{-6}$ .

## ELECTRICAL AND ELECTRONICS

### Ruthenium-Doped Semi-Insulator for Laser Diode

T. KITATANI *et al.* *U.S. Appl.* 2009/0,129,421

A semi-insulating layer is formed by doping a semiconductor material such as InP with Ru, Os, Rh or Ti, but in particular Ru. It is included between the p-type and n-type semiconductor layers to limit current leakage in the window region of a semiconductor laser, particularly a short cavity edge-emitting laser.

### Palladium Complex for Printed Circuits

HEWLETT-PACKARD DEV. CO, LP *U.S. Appl.* 2009/0,201,333

A Pd aliphatic amine complex (1) in a liquid carrier is inkjet printed on a substrate, a second composition containing a reducing agent such as formic acid is applied and the substrate is heated at 50–80°C to reduce (1) to Pd metal. Alternatively, (1) can be applied as a seed layer for a subsequent conductive metal such as Pt, Pd, Rh, Au, Cu, Ni or various alloys.

## PHOTOCONVERSION

### Luminescent Platinum Complexes

ARIZONA STATE UNIV. *World Appl.* 2009/086,209

Pt(II) di(2-pyrazolyl)benzene chloride and its analogs, which are obtained by forming an aromatic six-membered ring, 1,3-di-substituted by aromatic five-membered heterocycles such as pyrazolyl, imidazolyl, thiazolyl or substituted groups thereof, and reacting with an acidic Pt-containing solution. The benzene may be fluorinated, difluorinated, methylated, or replaced by pyridine. Cl may be replaced by a phenoxy group. The Pt(II) complexes, which in some embodiments are phosphorescent, are claimed for use as blue or white light emitters in OLEDs.

## REFINING AND RECOVERY

### Ruthenium Recovery from Solid Components

TOSHIBA KK *World Appl.* 2009/093,730

Ru is selectively recovered from hard disks, electrodes etc. by contact with an aqueous solution to form a Ru compound which is subsequently eluted and separated by filtration. The aqueous solution can be: (a) formic acid, (b) oxalic acid, (c) an acid and sugars, or (d) an acid and formic acid, alcohols, aldehydes or an acetal/hemiacetal compound (or precursors thereof). For (c) and (d), the acid is preferably 1–90 wt.% of the solution. The solid may also be oxidised by one of  $\text{O}_2$ , air,  $\text{O}_3$  or  $\text{H}_2\text{O}_2$  for second-pass removal of Ru.

### Dry Method for Recovering PGMs

DOWA METALS AND MINING CO LTD *Japanese Appl.* 2009-024,263

A sealed electric furnace is charged with spent pgm-containing solid components and granular Cu oxide having a particle diameter of 0.1–10 mm, a powdered reducing agent and a flux, and melting is done at pressures  $< 1$  atm. The pgms preferentially dissolve in the molten Cu, and the oxides are removed in the slag, which has final Cu content of  $< 3\%$ .