NEW PATENTS

ELECTROCHEMISTRY

Water Electrolysis Cell

HONDA GIKEN KK *Japanese Appl.* 2003-166,093

A water electrolysis cell (1) has a pair of catalytic layers (2) between which is a solid polymer electrolyte membrane. The catalyst in the anode side of (2) is made from a Ru-Ir alloy, and a metal(s) selected from Fe, Ni and Co, or an oxide of the alloy, or a mixture of alloy and oxide. At least one of Fe, Ni and Co has molar ratio w.r.t. Ru and Ir of 0.05–0.13 mol to 0.8–2.2 mol, preferably 1.8–2.2 mol of Ru and 0.8–1.2 mol of Ir. (1) gives a high energy efficiency over a long time.

PHOTOCONVERSION

Phosphorescent Dendrimers in LEDs

ISIS INNOVATION LTD *World Appl.* 03/079,736

An LED comprises at least one layer containing a phosphorescent organometallic dendrimer with a metal cation, such as Ir, and two or more coordinating groups (1) as part of its core. At least two of (1) each have a dendron attached, and at least one of them comprises at least one N atom forming part of an aromatic ring system or directly bonded to at least two aromatic groups.

Photoelectrochemical Cell

NIPPON SHEET GLASS KK *Japanese Appl.* 2003-163,037

An electrode for a dye sensitising-type photoelectrochemical cell of low internal resistance has a TiO_2 porous semiconductor body that includes a dye sensitising agent (1) and a conductive whisker (2). (1) is a bipyridine-carboxylic acid Ru complex hydrate and (2) is preferably of diameter 0.4–0.7 μm, length 5–20 μm, aspect ratio 10 to 20 and resistance 0.1–1 Ω cm^{-1}. The cell has little power generation loss.

Organic Electroluminescent Iridium Complex

MITSUBISHI CHEM. CORP *Japanese Appl.* 2003-192,691

An organic electroluminescent element (1) has a luminescent layer containing a mixed ligand-type organic Ir complex (2) sandwiched between an anode and a cathode. (2) gives improved colour purity in the emission of light of long wavelength, has high luminescent efficiency and gives long life to (1).

ELECTRODEPOSITION AND SURFACE COATINGS

Iridium-Aluminium Protective Surface Coating

GENERAL ELECTRIC CO *U.S Patent* 6,630,250

An article substrate is protected by a coating over its surface formed by depositing a layer of Ir, then a layer of Al on top of the Ir layer. The substrate, Ir layer and Al layer are then heated and form an Ir-Al protective coating. A ceramic thermal barrier coating may be applied over the protective coating.

APPARATUS AND TECHNIQUE

Biosensor for Detecting Macromolecular Biopolymers

INFINEON TECHNOL. AG *World Appl.* 03/079,016

Macromolecular biopolymers can be detected by a sensor that contains a unit to immobilise them and a unit to detect their signal. The latter unit uses a Pd, Pt, Au, etc., electrode. An evaluation circuit for the signal, containing a semiconducting layer with an organic material, is coupled to the detection unit.

Optical Sensor for Detecting an Analyte

METTLER TOLEDO GmbH *U.S Patent* 6,653,148

An optical sensor (1) for determining an analyte, specifically O_2, has a sensor matrix (2) made of a fluoropolymer. (2) contains a luminescent indicator with a metal complex of Ru, Rh, Ir or Re, and with at least one partially fluorinated ligand. Detection occurs by bringing (1) into contact with the measuring medium and by determining changes in an optical property resulting from interaction between the analyte and the luminescent indicator.

HETEROGENEOUS CATALYSIS

Catalyst for the Synthesis of Chiral Vicinal Diols

CSIR *European Appl.* 1,346,767

A multifunctional reusable catalyst M-(S), made on support (S) produces chiral vicinal diols by tandem and/or simultaneous reactions involving Heck coupling, N-oxidation and addition reaction of olefins in the presence of cinchona alkaloid compounds in a single pot. (S) is a layered double hydroxide (LDH), resin, SiO_2, etc., or unmodified support selected from resin and SiO_2. M is an active species of ≥ 2 of Pd, Ru, Os, W. M-(S) is PdOs-LDH, PdOsW-SiO_2, etc., and is used in place of soluble Os catalysts.

Treatment of Industrial Organic Pollutants

CNRS *World Appl.* 03/064,333

Industrial effluent containing organic pollutants is treated by ozonation in a reactor in the presence of a Ru and/or Ir catalyst (1) supported on CeO_2, ZrO_2 and/or TiO_2. Catalyst particle size is 20–500 μm. The effluent treated with ozone and the catalyst is continuously fed through a separation system to separate (1). Part of the effluent free of (1) is removed; the remainder is recycled with (1) into the reactor.

Production of Amines

BASF AG *World Appl.* 03/066,571

Amines are produced by the catalytic hydrogenation of nitroaromatics. The C supported catalyst of Pt, Pd and/or Ir is then separated from the reaction mixture which contains aromatic amine(s) and H_2O. The catalysts are separated by membrane filtration at 5–50 bar on the side of the suspension (1), a difference in pressure between (1) and the side of the permeate of ≥ 0.3 bar, and a flow rate of 1–6 m s^{-1} on (1).

Catalyst for the Reduction of NO to N_2

CSIC *World Appl.* 03/068,390

A catalyst (1) contains Pt crystals in contact with a mixture of MgO and CeO_2 or Pt/MgO-CeO_2 (2) which was selectively sulfated. (1) has excellent activity, selectivity and stability for reducing NO to N_2, using H_2 as a reducing agent, at 100–200°C and in an excess of O_2 (5 vol.%), H_2O (5 vol.%) and/or SO_2 (20 ppm). With (2), full NO conversion and 83% N_2 selectivity are obtained at 150°C with a reaction mixture of 0.25% NO/1% H_2/5% O_2/5% H_2O/He.

Production of 7-Alkyl-8-hydroxyquinolines

NISSAN CHEMICAL IND. LTD *World Appl.* 03/091,218

7-Alkyl-8-hydroxyquinolines (1) are produced in high purity and good yield by reducing 7-alkenyl-8-hydroxyquinolines under H_2 in the presence of an inorganic base, such as NaOH, and a Group VIII metal catalyst, such as Pd, Pd/C, etc. No substantial hydrogenation of the hetero-ring occurs so an adequate reaction rate can be obtained. (1) find use as an extracting agent for solvent extraction of metal ions.

Multiple-Component Metallic Combustion Catalyst

CLEAN DIESEL TECHNOL. INC *U.S. Appl.* 2003/0,148,235

Residual and lighter distillate fuels are combusted with greater efficiency using low concentrations of specific bimetallic or trimetallic fuel-borne catalysts, such as a fuel-soluble Pt with Ce and/or Fe catalyst (1). (1) reduces the fouling of heat transfer surfaces by unburned C while limiting the amount of secondary additive ash. Ultra-low levels of this nontoxic metal combustion catalyst are used to improve heat recovery and lower emissions of the pollutants.

Palladium and Zirconia Catalyst

CATACEL CORP *U.S. Patent* 6,620,761

A catalyst which includes Pd and Zr is formed as a suspension (1) and can be coated onto a metal strip. (1) is formed by combining Pd nitrate with a solution of an alkyl ammonium hydroxide, and further mixing with a hydrous Zr oxide. HNO_3 may be added to the Pd nitrate. The resulting composition exhibits superior activity and good adhesion to the metal strip, and can catalyse a combustion reaction as well as a steam reforming reaction.

Hydrogenolysis Catalyst

NE CHEMCAT CORP *U.S. Patent* 6,624,112

A hydrogenolysis catalyst (1) comprises: (a) a component(s) selected from Pd oxide, Pd oxide monohydrate, and Pd hydroxide in the Pd(II) oxidation state and contains 0.1–50 wt.% Pd, and (b) a component(s) from Pt, Ru, Rh, Ir and Au, on a non-organic porous support, such as activated C, acetylene black, SiO_2/Al_2O_3, TiO_2 and ZrO_2. (1) has high hydrogenolysis performance at low temperatures and during the debenzylation reaction of a compound wherein a benzyl group combines with a N atom. This is difficult for conventional catalysts.

Three-Way Catalysts

JOHNSON MATTHEY PLC *U.S. Patent* 6,625,976

A Pt group metal (pgm) three-way catalyst contains a high temperature support (1) and a low temperature support (2), both with enhanced catalytic activity at > 500°C and 200–400°C, respectively, and each being in the same washcoat layer. The mean particle size of (1) and (2) in the washcoat slurry is < 20 μm to prevent formation of a solution or a sol with the liquid medium of the slurry. The pgm is impregnated into the support either after formation of the washcoat on a non-porous refractory, metallic or palletised substrate, or before forming the washcoat slurry.

HOMOGENEOUS CATALYSIS

Manufacture of Ketopantolactone

ROCHE VITAMINS AG *World Appl.* 03/091,235

A process is described to oxidise pantolactone to ketopantolactone (1) with a periodate in the presence of a Ru catalyst in aqueous solvent using a microwave field. (1) is a key intermediate in the manufacture of pantothenic acid, a member of the B complex vitamins and a constituent of coenzyme A. The asymmetric hydrogenation of (1) yields D-(-)-pantolactone for pantothenic acid manufacture.

Hydrogenation of Carboxylic Acids

DAVY PROCESS TECHNOL. LTD *World Appl.* 03/093,208

A homogeneous process for the hydrogenation of carboxylic acids and/or their derivatives in the presence of a catalyst comprising Ru, Rh, Os, Pd or Fe and an organic phosphine is described. The hydrogenation is carried out in the presence of at least ~ 1 wt.% of H_2O. The catalyst can be regenerated in the presence of H_2 and H_2O.

Osmium-Assisted Oxidative Cleavage of Olefins

MICHIGAN STATE UNIV. *U.S. Appl.* 2003/0,149,299

An Os-assisted process for oxidative cleavage of oxidisable organic compounds, such as unsaturated organic compounds: alkenes and olefins, uses an Os catalyst chosen from OsO_4, $OsCl_3$, $K_2OsO_4{\cdot}2H_2O$, and a peroxy compound selected from peroxymonosulfuric acid and its salts. The C-C double bond of an organic compound can be oxidised to aldehyde, carboxylic acid, ester, etc., in a reaction that gives ozonolysis results but fewer problems. An aldehyde can thus be oxidised alone or with the Os in an interactive solvent to produce an ester or a carboxylic acid.

Ruthenium Metal Alkylidene Complexes

CALIFORNIA INST. TECHNOL. *U.S. Patent* 6,624,265

Ru alkylidene complex $(PCy_3)(L)Cl_2Ru(CHPh)$ catalysts, where L is a triazolylidene ligand, are more active for olefin metathesis at elevated temperatures than the parent $(PCy_3)_2Cl_2Ru(CHPh)$ (1). The complex with L = 1,3,4-triphenyl-4,5-dihydro-1*H*-triazol-5-ylidene (2) catalyses the RCM of substituted dienes to give tetrasubstituted cyclic olefins in good yield. (2) has the same stability towards O_2 and moisture as (1).

Carbonyl Compound Production

MITSUBISHI CHEM. CORP *Japanese Appl.* 2003-171,372

A carbonyl compound is produced by the dehydrogenation of an alcohol in the presence of an organic phosphine-Ru complex catalyst. The catalyst is obtained by mixing an organic phosphine and a Ru compound in the atomic ratio P : Ru of 2–8, followed by heating. Addition of a H acceptor is not needed. Both the reaction rate and reaction efficiency are increased.

FUEL CELLS

Aerogel Pt-Ru-C Catalyst in DMFC

KOREA INST. SCI. TECHNOL. *U.S. Appl.* 2003/0,176,277

An aerogel-type catalyst (1) contains 5–70 wt.% Pt and Ru, remainder C. The Pt:Ru atomic ratio is 1:4 to 4:1. A DMFC that employs (1) as an anode catalyst is disclosed. (1) is manufactured via a sol-gel process by adding metal salts and a base catalyst to a solution of organic gel materials, further adding a solution of basic amine and adjusting the pH to form a sol. This is followed by ageing, supercritical drying to make a type of aerogel (2), carbonising (2) and H reduction.

Nano-Level Platinum/Carbon Electrocatalyst

T. LU, X. LI and W. XING *U.S. Appl.* 2003/0,224,926

A nano-level Pt/C electrocatalyst (1) for the cathode of fuel cells is prepared using the chloride, bromide or iodide of NH_4 or K as the anchoring agent for the H_2PtCl_6. The Pt particles are homogeneously distributed in the interstices and on surfaces of the active C. (1) has high specific activity per unit mass for the catalytic reduction of O_2.

Cell Unit of a Fuel Cell

SANYO ELECTRIC CO LTD *Japanese Appl.* 2003-151,577

A cell unit of a fuel cell has a cell structure consisting of a Pt catalyst layer (1) and a gas diffusion layer arranged, in that order, on both sides of an electrolyte layer (2). (2) consists of a proton-conductive gel and an interlayer (3) containing C particles and electrolyte material. The porosity of (3) is smaller than that of (1). Even when (2) is formed using sol-gel processing, the pores in the catalyst layer are not covered by the electrolyte material. Pt utilisation in the catalyst is therefore increased and a high battery performance is obtained since the reaction gas is being fully supplied to the catalyst layer.

Fuel Cell Catalyst

HONDA GIKEN KOGYO KK *Japanese Appl.* 2003-173,787

A fuel cell, with an ion exchange membrane arranged between a negative and a positive electrode, generates power by contact of H_2 with the fuel cell catalyst contained in the negative electrode. At the same time O_2 contacts the catalyst in the positive electrode. The catalyst contains Pt alloyed with Fe, Co and/or Ni or other elements from Group VIII and Al. The catalyst can be easily structured and its catalytic function is as high or higher than that of Pt.

ELECTRICAL AND ELECTRONIC ENGINEERING

Multilayer Perpendicular Media

SEAGATE TECHNOL. LLC *World Appl.* 03/083,839

C or B is added into the CoCr layers of a multilayer perpendicular magnetic media (1) structure to reduce media noise. The structure of (1) has sharp interfaces between the Co-alloy layers and Pd or Pt layers, and significantly reduces exchange coupling. This relates to magnetic recording, especially to multilayer media that have columnar microstructure, and provides improved exchange decoupling and reduced noise.

Ruthenium Metal Layer

MICRON TECHNOL. INC *U.S. Patent* 6,617,248

A Ru metal layer for semiconductors is prepared by combining a Ru precursor with a measured amount of O_2 to form a Ru oxide (1) layer. (1) is annealed in H_2-rich gas to react the O in (1) with H_2. By varying the O_2 flow rate during the formation of (1), a Ru metal layer can be formed with various degrees of smooth and rough textures. The method reduces problems associated with manufacturing semiconductor devices, particularly in forming a Ru metal layer.

Synthetic Free Layer Structure for MRAM Devices

MAXTOR CORP *U.S. Patent* 6,649,960

A MRAM cell includes: a bottom electrode layer, a magnetic reference layer (1), an insulating layer, a synthetic free layer (2), and a top electrode layer. (2) has a first magnetic layer (3), a Ru antiferromagnetic coupling layer, and a second magnetic layer (4). (1), (3) and (4) are made of magnetic material, such as CoFeB, CoFe, or a bilayer of NiFe and CoFe. Layer (3) is made thicker than layer (4) for better operation.

Manufacture of Semiconductor Devices

HITACHI LTD *Japanese Appl.* 2003-158,203

A method for manufacturing a semiconductor device that can etch a Ru film (1) with good reproducibility and at the same time achieve the high etching selection ratio of a resist film is claimed. The method comprises forming a resist pattern on (1), then irradiating its surface with an UV ray in a pretreatment chamber within an etching unit to remove any organic contaminant adhered to its surface. (1) is then etched, using the resist pattern as a mask, in an ozone atmosphere inside the etching unit.

Capacitor for a Semiconductor Device

HYNIX SEMICOND. INC *Japanese Appl.* 2003-163,284

A semiconductor device includes a diffusion barrier layer (1) containing a ternary system of Ru, Ti and O formed by MOCVD on a substrate. A capacitor formed on (1) includes a bottom electrode formed on (1), a dielectric layer (2) formed on the bottom electrode, and a top electrode formed on (2). (1) prevents capacitor malfunction by supressing O diffusion.