NEW PATENTS

ELECTROCHEMISTRY

Durable Electrode for Electrolysis

ISHIFUKU MET. IND. CO LTD *Japanese Appl.* 2003-293,196

An electrode (1) is provided with: (a) a substrate of Ti or a Ti alloy; (b) an intermediate layer of: a Ti-Ta alloy layer, a porous Ta layer, and a layer of mixed Ir oxide and Ta oxide; and (c) an external layer of 50–98 mol% Ir oxide and 2–50 mol% Ta oxide. (1) has sufficient durability even when used as an anode for high speed plating of metal at high current density.

ELECTRODEPOSITION AND SURFACE COATINGS

Palladium Plating Solution

KOJIMA CHEM. CO LTD *European Appl.* 1,396,559

A Pd plating solution (1) contains: 0.1–40.0 g l^{-1} Pd; pyridine carboxylic acid and/or soluble Fe, Zn, Th, Se and/or Te salts; an amine derivative of pyridine carboxylic acid; an aldehydebenzoic acid derivative; and an anionic surfactant or an ampholytic surfactant. (1) can form high-purity stable Pd film deposits, thickness of 5 μm, which are free from cracks.

Electroless Platinum-Rhodium Alloy Plating

HONEYWELL INT. INC *U.S. Patent* 6,706,420

An electroless plating composition comprises an aqueous solution consisting essentially of: (a) a water soluble Pt nitrite salt or Pt ammine-nitrite salt; (b) a water soluble Rh nitrite salt or Rh ammine-nitrite salt; (c) ammonium hydroxide; and (d) hydrazine hydrate. A uniform coating of a Pt-Rh alloy can be deposited on virtually any substrate and material, including fibres and powders, of any geometrical shape.

APPARATUS AND TECHNIQUE

Detection of Oxygen Concentration in Exhaust Gas

ROBERT BOSCH GmbH *World Appl.* 03/106,989

A sensor element is claimed for determining the O_2 concentration in the exhaust gas of ICEs, in particular for a broadband lambda probe. It comprises: a solid electrolyte, which forms a pump cell; and a catalyst comprising two electrically connected electrodes, of Pt, Rh, Pd and/or their alloys, in an antichamber in the electrolyte. Measuring inaccuracies even with very high quantities of hydrocarbons are prevented.

Preparation of Noble Metal Nanotubes

JAPAN SCI. TECHNOL. CORP *World Appl.* 04/005,182

A nanotube (1) has a skeleton comprising: a single metal element (Pt, Pd, Rh, Ir, Au or Ag) or a mixture of two or more in an arbitrary ratio, with Ru or a base metal. (1) has an outer and an inner diameter, of ~ 5–7 nm and ~ 2–4 nm, respectively; a thickness of ~ 1–2 nm and a length of ≥ 10 nm. (1) are formed by admixing nonionic or ionic surfactants of different sizes, and then reducing the metal.

Photovoltaic Cell Interconnection

KONARKA TECHNOL. INC *U.S. Patent* 6,706,963

A photovoltaic module (1) with improved cell interconnections comprises a photosensitising agent of a Ru- or Os-complex, and an Fe complex. (1) includes a plurality of photovoltaic cells each having a photosensitised nanomatrix layer and a charge carrier "media". Preferably, the cells further include a catalytic "media" layer of Pt. The photovoltaic cells are disposed between two electrical connection layers.

Fructose Concentration Sensor

TAMA TLO KK *Japanese Appl.* 2003-227,811

A fructose concentration sensor comprises a Au electrode where fructose dehydrogenases is immobilised by a combination of Au and cysteamine, a Pt counter electrode, a Co phenanthroline complex (1) solution, and a Ag/AgCl reference electrode. (1) can be easily adjusted and reversible oxidation reduction can occur at relatively low potential. The sensor has high selectivity and sensitivity.

Oxygen Sensor Element

KYOCERA CORP *Japanese Appl.* 2003-315,303

An oxygen sensor element (1) comprises: a sensor section with a reference- and a measuring-electrode(s) made of Pt formed on opposing surfaces of a long ZrO_2 solid electrolyte plate; and a heater section with a heating element embedded in a ceramic insulating layer. (1) has excellent gas responsiveness and is capable of raising a temperature rapidly, while preventing the breakage of the element.

HETEROGENEOUS CATALYSIS

Three-Way Catalyst with NOx Storage Component

JOHNSON MATTHEY PLC *World Appl.* 03/100,228

A spark engine comprises an exhaust system with a three-way catalyst (TWC) containing Pt, Pd, Rh, Ru, Os and/or Ir; a NOx storage component of an alkali metal (K or Cs), an alkaline-earth metal (Mg) or a rare-earth metal (La, etc.); and an engine control unit to control the air:fuel ratio (1) of the engine. The amount of NOx contacting the TWC during lean running operation is determined by response to data input from a sensor to indicate the remaining NOx storage capacity (2) of the TWC. (1) is returned to stoichiometry when (2) is below a predetermined value, the arrangement being such as to prevent more NOx entering the atmosphere.

Platinum-Rhenium-Tin Catalyst

E. I. DU PONT DE NEMOURS CO *U.S. Patent* 6,670,490

An improved hydrogenation catalyst (1) comprises 0.5–3% Pt, 1–10% Re and 0.1–5% Sn supported on C, based on total weight of (1). (1) is used for hydrogenation of an hydrogenatable precursor in an aqueous solution, especially to produce tetrahydrofuran and 1,4-butanediol at 150–260°C.

Platinum Metal Catalysts by Immersion Coating

BASF AG *U.S. Patent* 6,676,919

Pt metal catalysts (1) are prepared by immersion coating a metallic support with at least one Pt metal complex. An aqueous medium which comprises Pt metal complex(es), reduction agent(s) and complexer(s) with pH > 4 is brought into contact with the metallic support to deposit the Pt metal as discreet, immobilised particles. The platinum metal comprises 80–100% wt.% Pd and 0–20% wt.% Pt or Ir. (1) are used for producing H_2O_2 or for hydrogenating organic compounds.

Three-Way Catalyst

JOHNSON MATTHEY PLC *U.S. Patent* 6,680,036

A three-way catalyst for vehicles contains an oxygen storage component comprising a mixed oxide (1) of Mn:Zr with molar ratio of oxides of 50:50–70:30 and surface area < 10 $m^2 g^{-1}$. (1) is obtained by co-precipitation, sol-gel or gel precipitation. The catalytically active metal is Pt, Pd and/or Rh. (1) can also contains a dopant selected from ceria and the oxides of Nd, Pr, La, etc. (1) gives improved capacity even after exposure to high temperatures.

Fischer-Tropsch Catalyst Enhancement

EXXONMOBIL RES. ENG. CO *U.S. Patent* 6,706,661

Both the activity and the CH_4 selectivity of a dispersed active metal (DAM) hydrogenation catalyst are enhanced by low temperature oxidation in a slurry phase forming a stable, unique oxidised catalyst precursor (1). This is subsequently reduced by treatment with H_2 at elevated temperature. Reducible promoters of Ru, Pd, Re, Fe and/or Co are mixed with (1) as a solution of their reducible salts. (1) are recovered from the mixture and treated with H-containing gas to simultaneously form the metals and reactivate the DAM hydrogenation catalyst.

Combustion Improvement Device for Petroleum Fuel

KANTAMU KK *Japanese Appl.* 2003-227,422

A combustion device noticeably decreases a toxic substance contained in an exhaust gas by improving the combustion of petroleum fuel by reforming physical properties of the petroleum fuel. A magnetism generating device is disposed on a sheet (1) carrying Au micropowder and a Pt catalyst (2) micropowder in a semiconductor material. Magnetic flux is released through (1). (2) contains Pt, Cu, Co, Mo and/or Yb.

Dehydrogenation Catalyst for Alicyclic Compounds

OSAKA GAS CO LTD *Japanese Appl.* 2003-320,251

A dehydrogenation catalyst (1) for an alicyclic compound contains a fibrous activated C (2) and at least one metal selected from Pt, Pd, Rh, Ir, Ru, Ni, Co, Fe, Cu, Ag, and Au. (2) has a specific surface area of at least 600 $m^2 g^{-1}$, an entire fine pore volume of at least 0.2 $cm^3 g^{-1}$, and an average fine pore diameter of 10–70 Å. (1) has a high activity and is capable of promoting the dehydrogenation reaction of an alicyclic compound at low temperatures.

HOMOGENEOUS CATALYSIS

Rhodium-Catalysed Hydroformylation of Olefins

OXENO OLEFINCHEMIE GmbH *World Appl.* 03/095,406

Aldehydes and alcohols are produced by the Rh-catalysed hydroformylation of olefins having 6–20C atoms. The discharge of the hydroformylation reaction is subsequently separated by distillation into the hydroformylation products and a solution containing Rh. The latter is redirected into the hydroformylation reaction. The Rh concentration of the redirected solution is 20–150 mass ppm.

Cross-Coupling of Alkyl(dialkylphenyl)indenes

BOULDER SCIENTIFIC CO *World Appl.* 03/101,601

A cross-coupling synthesis of 2-alkyl-4-(2,6-dialkylphenyl)indenes comprises treating a 2-alkyl haloindene with a 2,6-dialkylboronic acid in non-interfering hydrocarbon solvent. A cross-coupling catalyst (1) containing $PdCl_2$ and 1,5-cyclooctadiene is present. (1) improves an aryl chloride transformation.

Process for Conjugating C=C Double Bonds in Oils

ARCHER-DANIELS-MIDLAND CO *World Appl.* 04/016,350

A process for conjugating organic compounds (1) containing methylene interrupted C=C, such as triglyceride oils (linseed, soybean, sunflower, fish oils, etc.) comprises: solubilising Ru trichloride hydrate (~ 5–100 ppm based on the weight of (1)) with an organic solvent (monoalcohols or carboxylic acid) to form a first mixture. Then, further contacting this first mixture with (1) at a sufficient temperature and time to conjugate (1). This process can conjugate methylene interrupted C=C found in drying and semi-drying oils.

Preparing Oxirane Organosilicon Compositions

GENERAL ELECTRIC CO *U.S. Patent* 6,706,840

An organosilicon composition is prepared from an olefin and a SiH with a hydrosilation catalyst PtL_2X_2. X is chloride, bromide and iodide, in an amount of < 1 ppm based on the weight of the product. L is triphenylphosphine, etc. The method is useful in lowering the cost, coloration, and stability of the product, particularly when an oxirane-containing olefin is used in the hydrosilation. No inhibitor is needed to prevent undesired polymerisation of oxiranes in the reaction, and no product purification is required after removal of volatile components. The cured oxirane-containing organosilicon composition functions as an LED.

Triarylamine Production

HODOGAYA CHEM. CO LTD *Japanese Appl.* 2003-226,674

A *tert*-triarylamine (1) is produced by subjecting a diarylamine bearing 1–4 secondary diarylamino groups in one molecule and an aryl iodide to a condensation reaction in the presence of Pd acetate, tricyclohexylphosphine and Na or K *tert*-butoxide as the catalyst system, at 0–150°C for 1–24 h. (1) are selectively produced without forming byproducts. There is no restriction in manufacturing and handleability. (1) is a raw material for electronic products.

Optically Active 3-Quinuclidinol

KAWAKEN FINE CHEM. CO LTD

Japanese Appl. 2003-277,380

An optically active 3-quinuclidinol (1) is produced by the hydrogenation of 3-quinuclidinone in the presence of an optically active bidentate phosphine ligand and an optically active Ru(II) complex (2). (2) contains an optically active 1,2-ethylenediamine type ligand (with H or an alkyl group; and alkyl, aryl or aralkyl group which may have a substituent group, and two of which may form an alkylene) and a base. A highly optical isomer of (1) is obtained using an enantioselectively reducible (2).

Polymerisation Initiator System

KURARAY CO LTD *Japanese Appl.* 2003-321,509

A polymer of narrow molecular weight distribution is obtained by subjecting a radically polymerisable monomer (1) to living polymerisation. A polymerisation initiator of a transition metal complex with an electron donative group on the indenyl ring of a chloroindenylbis(triarylphosphine)Ru, and an organic halogen compound, are present. (1) is a methacrylic acid ester, etc. A side reaction is suppressed and the molecular weight is controlled.

FUEL CELLS

Hydrocarbon Reforming in Protonic Ceramic Fuel Cell

PROTONETICS INT. INC *World Appl.* 03/099,710

A process to convert hydrocarbons and H_2O vapour into H_2, CO and CO_2, and a fuel cell to produce electricity are claimed. The fuel cell comprises: a metallic and/or mixed conducting anode of metallic Pt, Ni alloy or a mixture of Ni oxide and oxide ceramic, capable of operating at < 850ºC; a cathode; and a proton-conducting ceramic electrolyte. Gaseous hydrocarbon fuels contact the anode; O_2 and H_2O vapour contact the cathode.

Gas Diffusion Layer for Fuel Cells

A. THOMPSON *et al.* *World Appl.* 04/004,054

A gas diffusion layer for a fuel cell is formed from a porous material comprising a solid matrix and interconnected pores, where part of at least one external surface is coated with an electrically conductive material (1) of resistivity < 20 Ω cm. (1) are metals, such as Pt, Au, Ni, Co, etc., or their alloys, etc. They may be applied to the foam strands by electroplating, electroless plating, sputtering, plasma vapour deposition, etc.

Devices Containing Platinum-Iridium Films

SYMYX TECHNOL. INC *U.S. Patent* 6,682,837

The electrochemical conversion of a hydrocarbon-based fuel (1) (such as MeOH) and O_2 to H_2O, CO_2 and electricity in a fuel cell (1) is claimed. (1) comprises: an anode, a cathode, a proton exchange membrane electrolyte, and an external circuit. (1) is contacted with a ternary metal alloy catalyst (in at. %): 25–50 Pt, 25–55 Ru, and 5–45 Pd, to oxidise the fuel. The difference between Ru and Pt is ≤ ~ 20 at. %.

ELECTRICAL AND ELECTRONIC ENGINEERING

Semiconductor Electronic Device

STMICROELECTRONICS SRL *European Appl.* 1,367,644

A semiconductor electronic device (1) comprises a die of a semiconductor material formed with a plurality of contact pads (2), electrically connected to a holder by wire leads. (1) comprises a welding stud bump containing Pd, Au, or their alloys, formed on each (2). (1) is highly reliable and can be fabricated simply at low cost.

Semiconductor Device

RICOH CO LTD *European Appl.* 1,385,218

A semiconductor device (1) able to increase the mobility of carriers and reduce the current in the OFF state is claimed. (1) includes a gate electrode (2), a first and a second electrode formed from Pd, Pt, Cr, Ta, etc., and an insulating layer of resin, etc., on (2). The first and second electrodes are on the insulating layer with an organic semiconductor layer (3) between. A first resistance layer comprises conductive polymers and has lower electrical resistance than (3).

Platinum-Cobalt Sputtering Targets

HERAEUS INC *European Appl.* 1,395,689

A Co-Cr-B-Pt sputtering target alloy having multiple phases can also include Cr, B, Ta, Nb, C, Mo, Ti, V, W, Zr, Zn, Cu, Hf, O, Si or N. The alloy is prepared by mixing Pt powder with a Co-Cr-B master alloy, ball milling followed by hot isostatic pressing to densify the powder into the alloy.

Dielectric Interconnect Frame

RAYTHEON CO *World Appl.* 04/013,934

A frame structure for a transmit/receive module (1) configured to transmit and receive electromagnetic radiation comprises a frame component (2) formed as a single piece from a synthetic resin dielectric material. (2) has a thin film coating including a Pd layer on top of a Ti getter layer to provide a ground connection and electromagnetic shielding when the frame structure is incorporated into (1). The synthetic resin dielectric material provides (2) with a range of compressibility that gives an effective ground connection. (1) is used for H_2 getters for GaAs hermetically-sealed packaging.

Contact Resistance Reduction in Organic FETs

INFINEON TECHNOL. AG *World Appl.* 04/017,440

Reducing the contact resistance in organic field effect transistors made with Pd contacts is achieved by layer(s) of either Pd(0) or Pt(0) phosphines. A first contact injects charge carriers into the semiconductor (1) and a second contact extracts charge carriers from (1). The phosphine layer(s) lie between the contacts and (1), and allow charge transfer between the first contact and the organic semiconductor material. The phosphine gives significantly reduced contact resistance between the contact and the organic material.

Devices Containing Platinum-Iridium Films

MICRON TECHNOL. INC *U.S. Patent* 6,660,631

Pt-Ir films (1), formed on semiconductor devices, such as capacitors, integrated circuit devices, memory cells, etc., are deposited by vaporising the precursor compositions (1) and directing them toward the semiconductor substrate by CVD. (1) comprises a Pt complex selected from $CpPt(Me)_3$ (Cp is substituted or unsubstituted cyclopentadienyl), $Pt(CO)_2Cl_2$, *cis*-$Pt(CH_3)_2[(CH_3)NC]_2$, $(COD)Pt(CH_3)_2$, etc.

Magnetic Recording Media with Ruthenium

SEAGATE TECHNOLOGY LLC *U.S. Patent* 6,680,106

The corrosion protection of magnetic recording media (1) is achieved by using: a thin protective barrier layer of Ru < 10 Å formed of elemental Ru, a Ru oxide and/or a Ru alloy containing 1–50 at.% of Ti, Mo, W, Nb, Ta, etc., on the magnetic layer. A C protective layer (10–50 Å in thickness) is then formed on the corrosion protective layer. (1) are used for drive programs with reduced flying height, or pseudo-contact/proximity recording.

Selective Formation of Top Memory Electrode

ADV. MICRO DEVICES INC *U.S. Patent* 6,686,263

Electroless plating for the formation of the top electrode of an organic memory device operates at relatively low temperatures (35–85°C). The electroless process is utilised to form conductive layers, such as electrodes and the like, from Pd, Pt, Ag, Ni, Co, Ti, Zn, etc., and includes depositing an activation compound, such as SnPd, on selected areas of conductive organic media. A chemical solution containing metal ions is then applied. The ions are reduced and are thus plated onto the conductive layer.

Plated Metal Transistor Gates

MOTOROLA INC *U.S. Patent* 6,686,282

Metal gates for N-channel and P-channel transistors are formed from a first and second metal layer, by plating with Ru, Ru oxide, Ir, Pd, Pt, Os, Ni, and Co, to achieve their appropriate work functions. The plating is achieved with a seed layer consistent with the growth of the desired layer. The metal layers are formed either by electroless or electrolytic plating with a Pt metal, W, Ru oxide, etc., and at least one refractory metal or Zr, Hf, La, Lu, Eu, etc.

Devices with Platinum-Rhodium Layers

MICRON TECHNOL. INC *U.S. Patent* 6,690,055

A capacitor for integrated circuits (ICs) comprises a first electrode, a dielectric layer of Ti_2O_5 and a second electrode, at least one of which consists of a single layer of a CVD Pt-Rh alloy. Pt-Rh barriers and electrodes for cell dielectrics for ICs, particularly for DRAM cell capacitors are also claimed. The Pt-Rh barriers protect surrounding materials from oxidation during oxidative recrystallisation steps and protect cell dielectrics from loss of O during high temperature processing steps. Plating a Rh-containing layer on a semiconductor wafer is also claimed.

Low Resistance Conductor Leads for GMR Heads

HEADWAY TECHNOL. INC *U.S. Patent* 6,706,421

A lead structure for use with a magnetoresistive sensing element in a magnetic disk system comprises a layer of Ru or Rh sandwiched between layers of a Ni-Cr alloy (1). The lower (1) layer acts as a seed layer to ensure that the Ru and Rh layers have crystal structures corresponding to low resistivity phases. The interfaces between these three layers introduce a minimum of interfacial scattering of the conduction electrons thus keeping dimensional increases in resistivity to a minimum.

Dye-Sensitised Metal Oxide Semiconductor

NATL. INST. ADV. IND. TECHNOL. *Japanese Appl.* 2003-272,721

A dye-sensitised metal oxide semiconductor electrode (1) uses a Ru(II) complex having: a bonding group selected from a carboxyl group, a sulfonic acid group, etc.; a diketonate; and a halide, a cyano group or the like. (1) in a solar battery is thermally and optically stable, and efficiently uses the energy in sunlight by absorbing light over a wide wavelength range.

Thick Film Circuit Board

DENSO CORP *Japanese Appl.* 2003-332,711

A Ru resistor (1) is formed on an insulating board in an atmospheric environment; a thick film Cu conductor is then baked onto it at low temperatures of 500–700°C so as to be electrically connected to (1). The thick film circuit board (2) so formed has improved conductor characteristics without (1) losing its resistance reliability. (2) can cope with increased wiring density, high frequencies and large currents.

MEDICAL USES

Sacrificial Anode Stent System

SCIMED LIFE SYSTEMS INC *World Appl.* 04/002,328

A sacrificial anode stent system comprises a stent with sacrificial anode portion(s) of Mg, Zn, Al, mild steel, low alloy steel, etc., at which corrosion can occur, and a vaso-occlusive device which includes a coil of Pt. The non-sacrificial portion of the stent includes stainless steel. The stent comprises radiopaque portions. The vaso-occlusive device has at least one portion with a potential different from that of the sacrificial anode portion.

High Specific Activity Platinum-195m

UT-BATTELLE LLC *World Appl.* 04/015,718

High-specific-activity ^{195m}Pt is produced by exposing ^{193}Ir to a flux of neutrons sufficient to convert a portion of the ^{193}Ir to ^{195m}Pt to form an irradiated material. The irradiated material is dissolved in *aqua regia* at ≥ 217°C to form an intermediate solution of Ir and Pt. The Pt is then separated from the Ir by cation exchange chromatography using HCl, thiourea, followed again by HCl. This method can prepare medically useful high-specific-activity radioisotopes, particularly ^{195m}Pt with activity ≥ 90 mCi mg^{-1}.