

NEW PATENTS

ELECTROCHEMISTRY

Gas Diffusion Electrode

DE NORA S.p.A.

European Appl. 931,857A

A gas diffusion electrode (1) comprises an electrically conductive web which is coated on at least one side with a Rh metal-Rh oxide catalyst supported on C black. A method for the preparation of (1) is also claimed. The electrode is used as the cathode in the production of Cl₂ from HCl acid. The catalyst has improved resistance to dissolution in highly corrosive environments, such as a highly oxidising HCl/Cl₂ mixture.

Sodium Persulfate Production

MITSUBISHI GAS CHEM. CO. INC. *German Appl.* 1/98/58,544

High purity Na persulfate is produced by electrolysis of an electrolyzing solution containing NH₄ sulfate, Na sulfate and Na persulfate and preferably Na ions, using a Pt anode operating at an anodic current density of < 40 A dm⁻² at 15–40°C. The resulting liquid is reacted with NaOH. The process allows the industrial manufacture of high purity, N-free Na persulfate in high yields and at high electrolysis current efficiencies. The Na persulfate is useful as a polymerisation initiator for polyvinyl chloride and polyacrylonitrile, and as an etching agent for treating printed circuit boards.

ELECTRODEPOSITION AND SURFACE COATINGS

Metal Plating of Polymers

E. I. DU PONT DE NEMOURS & CO. *World Appl.* 99/39,021A

Liquid crystalline polymers (LCP) are sputter coated or ion plated with Pd and subsequently electrolytically plated with, for example, Cu, at a current density ≥ 5 A dm⁻² and/or a rate of ≥ 1 $\mu\text{m min}^{-1}$, to make metal plated parts in which the metal has good adhesion to the LCPs. Before or after the electrolytic plating, the metal coating may be patterned. Electroconductive, optically reflective or decorative coatings may be formed. Parts having a patterned metal surface may be used in printed circuit fabrication.

Palladium Electroplating Liquid

MATSUDA SANGYO K.K.

Japanese Appl. 11/189,891

A Pd plating solution for lead frame, semiconductor devices and electronic components, includes 1–30 g l⁻¹ of pyridine sulfonic acid, or 0.1–20 g l⁻¹ of its salts and 0.1–300 ppm of soluble thallium salts. The Pd deposits have improved solder wettability and heat resistance.

Platinum Coating Method

TANAKA KIKINZOKU KOGYO K.K. *Japanese Appl.* 11/228,260

A Pt coating method for protecting Al₂O₃ crucibles (1) from corrosion by molten glass at high temperature involves thermal spraying and then peeling of a Pt film, followed by applying, drying and baking a Pt paste. A high quality surface layer of Pt without any holes is formed. This prevents molten glass from soaking into (1).

APPARATUS AND TECHNIQUE

Photocatalyst Filter

EQUOS RES. K.K.

Japanese Appl. 11/151,406

A photocatalyst filter for removing harmful formaldehyde and NO_x, etc., from air, contains a Ti oxide-Pd composite catalyst with 0.05–1.5 wt.% Pd in the photocatalyst. Pulp, which can be formed into a sheet, is used as the substrate material, and 30–50 wt.% photocatalyst per pulp is uniformly incorporated in the substrate. To incorporate the Ti oxide-Pd composite catalyst into the pulp, the catalyst and the pulp are both dispersed in H₂O.

Hydrocarbon Gas Sensor

TOYOTA CHUO KENKYUSHO K.K. *Japanese Appl.* 11/174,021

A hydrocarbon gas sensor has a Pt/Ag cathode and an anode, with the volt-ampere characteristic set so that the gas sensing element exhibits negative differential resistance. Measuring hydrocarbon gas is accurate as its concentration is not influenced by O₂ gas concentration. The sensor has only one sensing element, so is reduced in size and weight and can be easily installed. It is used for detecting CH₄, for measuring the density of natural gas and for detecting various gas components released from I.C.E., motor vehicles, boilers, etc.

Esterified Fuel Manufacture

JATCO CORP.

Japanese Appl. 11/181,454

Apparatus for manufacturing esterified fuel has a "rotation feather" to stir a mixture of specific composition containing H₂O, edible oil, MeOH and KOH, and is actuated by a motor using the e.m.f. generated by a Pt electrode, a Mg electrode of differing ionisation, and the mixture contained in a reaction vessel. The apparatus heats and stirs the mixture using the generated e.m.f., and thus saves energy.

Detection of Combustible Gases

MATSUSHITA DENKI SANGYO K.K. *Japanese Appl.* 11/183,427

A gas sensor for detecting combustible gases, and also for CO, from the waste gas of petroleum combustion engines, has an oxygen ion conductivity electrolyte enclosed by a pair of Pt electrodes, which have an outside seal. A heater and a porous catalyst are also assembled on one side of a pair of ceramic gas-transmission materials. The ceramic, selective, gas-transmission material blocks acidic gas and prevents poisoning of the gas sensor.

Oxygen Sensor for Molten Metal

AGENCY OF IND. SCI. & TECHNOLOGY

Japanese Appl. 11/242,016

An O₂ sensor for molten metal, such as glass, for manufacturing liquid crystal substrates and optical glass, comprises a conducting Pt wire, inserted into a recess on a ZrO₂ rod of a reference electrode and attached to the Al₂O₃ tube of a measurement electrode. The O₂ activity is based on the potential difference generated between the two electrodes, and the result is unaffected by differences in thermal expansions of the Pt and zirconia rod.

HETEROGENEOUS CATALYSIS

Manufacture of Vinyl Acetate

CELANESE G.m.b.H. *European Appl.* 922,490-491A

A catalyst (1) for the production of vinyl acetate from ethylene, acetic acid and O₂ is obtained by impregnating a support with soluble Pd and Au compounds, followed by treatment with a peroxy compound, addition of an alkali metal compound and drying. Alternatively, the impregnated support can be irradiated with microwaves before, during or after one of the above steps. (1) has improved selectivity, gives less byproduct, especially CO₂, and has improved activity, thus permitting lower reaction temperatures for the same space-time yield.

Production of Unsaturated Cyclic Ethers

BASF A.G. *European Appl.* 933,374A

Unsaturated cyclic ethers are produced from diols in high yield and with low hydrogenated byproduct formation in the presence of a supported Co catalyst impregnated with Pt, Pd, Rh, Ir, Ru, Os and/or Re on an inert carrier doped with S, at 150–300°C. The catalyst has high and reproducible activity and produces 3,4-dihydro-2H-pyran with a low tetrahydropyran content.

3-Glycidyoxypropyltrialkoxysilane Production

HUELS A.G. *European Appl.* 934,947A

3-Glycidyoxypropyltrialkoxysilanes (1) are produced by hydrolysis between an allylglycidyl ether and a trialkoxysilane in the presence of a Pt/non-metallic support catalyst, easily prepared by impregnating or spraying with a solution of a Pt(II) or Pt(IV) compound, drying and reducing to Pt(0) in H₂. Selectivity is > 90% at 140°C and the reaction is easily controlled, with only small amounts of high and low boiling side products being formed. (1) are important intermediates and end products in organosilane chemistry, useful as adjuvants and binders in the lacquer, glass fibre, foundry and adhesive wax industries.

Alkane Dehydrogenation Catalyst

JOHNSON MATTHEY PLC *European Appl.* 937,697A

The dehydrogenation of an alkane to an alkene, especially isobutane to isobutene, is carried out in admixture with O₂ and in the absence of added steam over a dehydrogenation and oxidation catalyst comprising 0.1–3 wt.% of Pt deposited on a support of a mixture of Sn oxide and Zr oxide. The catalyst possesses good activity for the dehydrogenation of an alkane, with high selectivity and high yield of alkene.

Hydrodechlorination Catalyst

AKZO NOBEL N.V. *World Appl.* 99/17,877A

The durability and selectivity of a supported Pt group metals hydrodechlorination catalyst (1) can be improved by treating the supported catalyst with one or more of late or post-transition metal halide salts, such as Zn, Sn and Cu chlorides; and an alkali metal halide and/or NH₃ halide. (1) has a Pt(0) group metal component predominantly residing adjacent to the surface of the support in a form visible under a microscope of resolution ~ 5 Å. (1) may retain 90% conversion for ≥ 56 h, and has conversion selectivity for CCl₄ to CH₂Cl₂ of 80–92%.

Stable Calcined Catalyst

KTI GROUP B.V. *World Appl.* 99/29,420A

A calcined catalyst for the dehydrogenation of paraffinic hydrocarbons to the corresponding olefins has an oxidic, thermally stabilised support, a catalytically active constituent comprising 0.2–2.0 wt.% Pt and/or Ir and a promoter selected from (in wt.%): 0.2–5.0 Ge, Sn, Pb, Ga, In and/or Tl; 0.1–5.0 Li, Na, K, Rb, Cs and/or Fr; 0.2–5.0 Fe, Co, Ni and/or Pd; 1.0–5.0 P; 0.2–5 Be, Mg, Ca, Sr, Ba, Ra and/or lanthanides; and 0.1–2 Cl. The catalyst is highly efficient, with good conversion rates and selectivity, and high operating stability of relatively long cycle times before needing regeneration.

Exhaust Gas Cleaning Catalyst

TOYOTA JIDOSHA K.K. *World Appl.* 99/32,223A

An exhaust gas cleaning catalyst for I.C.E. and boilers comprises a colloid of a composite of noble metals, such as Pt and Rh, supported on a porous oxide carrier of particle size larger than the conventional ones. This lowers vapour pressure and inhibits the release and diffusion of volatile components, such as PtO₂. The affinity between the colloid and the carrier is improved, so noble-metal migration and particle growth are inhibited, as is S poisoning of the NO_x absorbent; catalytic activity is sustained and catalyst durability is improved.

Thermally Durable Three-Way Catalyst

JOHNSON MATTHEY PLC *World Appl.* 99/34,903A

A Pt group metal three-way catalyst (1) for treating vehicle exhaust gases comprises a high temperature catalyst support material, a low temperature catalyst support material and at least one Pt group metal, all in a layer on a non-porous support substrate, and an additive (2) effective at suppressing H₂S emissions. If (2) is in the layer, it is segregated from the other components. H₂S emissions are reduced without reducing thermal durability of the catalyst, even on extended high-temperature ageing.

Preparation of Betaines

AIR PROD. & CHEM. INC. *U.S. Patent* 5,895,823

Preparation of an aqueous solution of a betaine comprises reacting an aqueous solution of a choline hydroxide with O₂ in the presence of a supported Pt or Pd catalyst, such as Pt/C, at 20–100°C and pressure from atmospheric to ~ 100 psi. This process is advantageous over prior art processes because no amine halocarboxylate contaminants are produced and no H₂O₂ is generated. When choline salts which do not require added base are used, no alkali metal salts are generated. Betaines are used in animal feeds and skin cleansers.

Desulfurisation of Distillate Streams

EXXON RES. & ENG. CO. *U.S. Patent* 5,935,420

Hydrodesulfurisation of distillate streams, such as petrochemical or petroleum, which contain multiple condensed ring heterocyclic organosulfur compounds, involves contacting with Pt, Pd, Ir and/or Rh catalysts supported on inorganic refractory substrates under relatively mild conditions of 40–500°C and 100–3000 psig. The catalyst system also contains a H₂S sorbent.

Removal of Organic Sulfur Compounds

UOP L.L.C.

U.S. Patent 5,935,422

The removal of aromatic S compounds from a fluid catalytic cracking feedstock with minimal adsorption of aromatic hydrocarbons is achieved using a zeolite Y catalyst exchanged with alkali or alkaline earth cations, especially K-exchanged zeolite, impregnated with a 0.05–1 wt.% of zero-valent Pt or Pd. The S-laden adsorbent is effectively regenerated by heating in flowing H₂ at 25–500°C.

Preparation of Hexahydrophthalides

NEW JAPAN CHEM. CO. LTD.

Japanese Appl. 11/180,968

A preparation of hexahydrophthalides (1) comprises nuclear hydrogenation of phthalides under pressures of 1–80 kg cm⁻² and at 100–240°C in the presence of a Pd-type catalyst. The method is industrially advantageous as it can give (1) derivatives in good yields under comparatively low pressures. (1) are used as starting materials for pharmaceuticals, agrochemicals and paints.

Production of Secondary Amines

BASF A.G.

German Appl. 1/97/56,947

A process for preparing secondary amines, X(CH₂NHR)_n (1), where n = 1–4, R = 1–200C alkyl, 3–8C cycloalkyl, etc., including polyfunctionals, comprises reacting nitriles, X(CN)_n, with primary amines, RNH₂, and H₂ at 50–250°C and 5–350 bar in the presence of a catalyst of 0.1–10 wt.% Pd/support. Good selectivities (83–91%) with long catalyst life are reported. (1) are used as hardeners for epoxy resins, catalysts for polyurethanes and intermediates for quaternary NH₄ compounds, etc. Polyfunctional (1) are also useful for production of synthetic resins, ion exchangers, pharmaceuticals, plant protection agents and pesticides.

Three-Way Catalyst Production

HYUNDAI MOTOR CO. LTD.

German Appl. 1/98/3,799

A three-way catalyst is produced by reducing an Al₂O₃-containing Pd solution, adding CeO₂ and a mixed solution to this solution, adding acetic acid to the reaction solution and adjusting the pH to < 4.5. The mixture is then ground, so that 90% of the particles have diameter 7–9 μm, dried and calcined onto a ceramic support. The resulting catalyst is effective at removing pollutants, such as hydrocarbons, CO and NO_x from vehicle exhaust gases.

HOMOGENEOUS CATALYSIS

Telomerisation of Butadiene

BAYER A.G.

European Appl. 939,074A

Two-phase telomerisation of butadiene with NH₃ in the presence of a hydrophilic Pd complex allows separation of products octa-2,7-dienyl-1-amine (1) and octa-1,7-dienyl-3-amine (2) and the isomerisation of (2) to give (1). AllyldiaminoPd(II)-tetrafluoroborate, used in the preparation of the Pd complexes with phosphantriy benzenesulfonic acids, is claimed. (1) can be obtained in high yields at high selectivity and is useful as an intermediate for octylamines, which can be used in corrosion inhibitors, emulsifiers, etc.

Manufacture of 2,6-Naphthalenedicarboxylic Acid

EASTMAN CHEM. CO.

World Appl. 99/29,649A

A process is claimed for the manufacture of 2,6-naphthalenedicarboxylic acid (1) by the liquid phase oxidation of 2,6-dimethylnaphthalene with a O₂-containing gas in the presence of a catalyst system comprising Co, Mn, Br and Pd. The presence of Pd during the oxidation process permits the production of (1) in good yields and rates, and gives lower production rates of trimellitic acid. (1) is used as a monomer for the production of several high performance polyesters, such as liquid crystal polyesters and polyamines.

Aminopropylmethylsiloxanes

PCR INC.

U.S. Patent 5,892,084

High purity 3-aminopropylmethylsiloxanes (1) with isomeric purity of > 85% were prepared by hydrosilylation of an allylamine and a monohydridomethylsiloxane in the presence of a neutral Pt catalyst. The Pt catalyst is present in an amount of 5–150 parts by weight of Pt per 10⁶ parts of the combined weight of monohydridomethylsiloxane and allylamine. (1) are formed in higher isomeric purity than with alternative methods. (1) are fluids used as intermediates in the preparation of organofunctional siloxanes for use in cosmetics, textiles, coatings and adhesives.

Herbicidal Arylamide Derivatives

LONZA A.G.

U.S. Patent 5,900,484

Arylamides derivatives (1) of heteroaromatic carboxylic acids are prepared from the corresponding heteroaromatic halogen compounds, the corresponding aromatic amines and CO in the presence of a Pd complex with triphenylphosphine and with a base. A Pd complex of *para*-substituted triphenyl gives greater yields than *meta*-, *ortho*- or unsubstituted complexes. The yield is almost quantitative and (1) have activity as herbicides.

Amino Acetylenes

SECIFARMA S.p.A.

U.S. Patent 5,902,902

Preparation of 3-aminophenylacetylene derivatives comprises reacting a 3-haloaniline with an acetylene derivative in the presence of Pd and Cu complexes, such as Pd(OAc)₂, Pd tetrakis(triphenylphosphine) and CuCN, and a strong organic base, optionally followed by removal of the protective group. The process is highly selective, simple, uses readily available starting materials, involves fewer steps and has improved safety than the prior art. Amino acetylenes are useful as antithrombotic, anti-inflammatory and anticancer agents.

Manufacture of Alkanediolen

MITSUBISHI CHEM. CORP.

Japanese Appl. 11/189,557

A reaction mixture containing conjugate alkydiene and H₂O is reacted in the presence of CO₂ and a catalyst solution containing Pd and organic P compound. The catalyst solution is extracted with H₂O in the presence of an amine, and organic acid byproduct is removed. The catalyst solution is recirculated and deactivation of the circulating catalyst is prevented. The amount of waste H₂O requiring treatment is much reduced since the amine and H₂O are recovered.

FUEL CELLS

Fuel Cell Catalyst

TOYOTA JIDOSHA K.K.

World Appl. 99/16,706A

A catalyst for the selective oxidation of CO in a H₂-rich gas comprises: Ru as a main component supported on a carrier, an alkali metal, such as Li, and/or an alkaline earth metal, which have the effect of increasing the temperature range for promoting the selective oxidation. An apparatus containing the catalyst for high level selective oxidation is claimed. The catalyst is used for fuel cells, especially for powering electric automobiles.

Anode Electrocatalyst for a Fuel Cell

STONEHART ASSOC. INC.

U.S. Patent 5,922,487

An anode electrocatalyst for a fuel cell comprises an alloy of 33–55 at.% Mo and Pt, with a particle size of 10–60 Å. The electrocatalyst is carried on a support having a surface area of 60–2000 m² g⁻¹. The catalyst has excellent anti-poisoning characteristics, thus allowing the use of a reforming gas in a solid PEFC, which would otherwise be liable to be poisoned.

Fuel Cell Oxidation Catalyst

NISSAN MOTOR CO. LTD.

Japanese Appl. 11/114,423

A catalyst (1) for the selective oxidation of CO in H₂ gas comprises a complex oxide of a base metal, such as Cu, containing Pd and/or Ru metal and rare earth metal dispersed in activated Al₂O₃ and coated on the surface of a ceramic integrated carrier. The reduction of the complex metal oxide to its metals is prevented and catalyst deterioration is reduced. (1) is used for a fuel battery in an electric power generator, car battery system, etc.

Water-Soluble Metal Colloid Solution

HOECHST A.G.

German Appl. 1/97/45,904

A H₂O-soluble metal colloid solution (1), used as a catalyst for fuel cells and electrolysis cells, comprises Pt compound(s) and optionally Rh, Ru, Ir or Pd compound(s), and is stabilised using a proton-conducting protective colloid. The preparation of (1) using a reducing agent and a cation exchange polymer as stabiliser is also claimed. (1) is stable, with no tendency to recrystallise on touching the membrane in membrane-electrode units.

ELECTRICAL AND ELECTRONIC ENGINEERING

Large Oxide Superconductor Body

INT. SUPERCOND. TECHNOL. CENT. *European Appl.* 890,661A

A large-sized oxide superconducting bulk body (1) with excellent characteristics and high homogeneity, suitable as a strong magnetic-field magnet, is prepared by a seed crystal method. A precursor is prepared by press-moulding powder obtained by mixing REBa₂Cu₃O_{7-x} with RE₂BaCuO₅ or RE₂Ba₂Cu₃O₁₀ (RE = rare earth metal) powder and a Pt additive. The precursor is homogeneously semi-melted at a holding temperature for a prescribed time, and is crystal-grown at a temperature lower than the melting point. (1) are used as superconducting magnets, flywheels for energy storage, carrying devices, bearings, etc.

Conductive Fluororesin

SHINETSU CHEM. CO. LTD.

European Appl. 923,084A

A conductive fluororesin composition (1) used to bond semiconductor devices and to mount quartz crystal oscillators comprises: a reactive fluorinated polyether compound with at least two aliphatic unsaturated hydrocarbon radicals per molecule, a compound with ≥ 2 H atoms, each directly attached to a Si atom, a Pt group metal catalyst and Ag particles. (1) cures into rubbery parts with improved heat-, weather- and solvent-resistance, and can form strong bonds to various substrates.

Lead Frame for Semiconductor Devices

S. SERIZAWA

Japanese Appl. 11/111,909

A lead frame for a semiconductor device has a pair of Pd, Au or Cu layers (0.005–0.1 μm in thickness), a Ag plating layer (0.005–5 μm), and a Pd or Pd alloy plating layer (0.005–1 μm), sequentially formed on the upper and lower surfaces of a lead frame material. The formation of Pd oxides is reduced and diffusion of Pd into the Ni base is prevented. Both wire bonding and solder wettability are improved by the thin Pd plating layer.

Dielectric Capacitor

SONY CORP.

Japanese Appl. 11/121,702

A thin film material suitable for the lower electrode material of a dielectric capacitor (1) comprises two thin films having (in at.%): (a) Pd_a(Rh_{100-x-y-z}Pt_xIr_yRu_z)₁₀₀O_c, where a = 20–70; b = 10–40; c = 15–60; a+b+c = 100; x = 0–100; y = 0–100; z = 0–100 and x+y+z = 0–100, and (b) Ir_pPt_qRu_r, where p = 0–100; q = 0–100; r = 0–100 and p+q+r = 100. A dielectric film is formed on the lower electrode by CVD. The thin material gives dielectric film with a good surface and the material can be used in ferroelectric nonvolatile- or semiconductor-memories.

Non-magnetic Foundation Film

SHOWA DENKO K.K.

Japanese Appl. 11/195,530

A non-magnetic foundation film (1) is formed from alloy of formula (Ni,Al_{1-x})_{100-y-z}M_y, where y is 0.4–0.6. M includes (in at.%): 1–40 Cr, 1–50 Mo, 1–60 W, 1–35 V, 1–25 Zr, 1–35 Nb and 1–25 Pd. Magnetic film consists of Co alloy formed on the foundation film. (1) is used in magnetic tape, disk and drum; (1) reduces noise in magnetic recording media and improves output of the magnetic recording medium by increasing coercive force.

TEMPERATURE MEASUREMENT

Temperature Sensor

MURATA MFG. CO. LTD.

Japanese Appl. 11/121,208

A temperature sensor (1) for use in motor vehicles has a heat resistant insulated substrate having surface roughness of 0.5–5 μm on an Rz display. A Pt film is formed on the substrate by electroless plating. Contact between the Pt film and the surface is highly improved and Pt loss from the surface is prevented, enabling (1) to be used at temperatures of 1000°C.

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