

NEW PATENTS

ELECTROCHEMISTRY

Oxygen Generating Electrode

TDK CORP. *European Appl.* 699,780A

An O₂ generating electrode of good service life and strength comprises: a conductive substrate coated with mixed Ir/Ta oxide layers; the second and third layers contain 80–99.9 and 40–79.9 mol% Ir, respectively, as a repeat unit, and a first layer of 14–8.4 mol% Ir, remainder Ta. The electrode has low O₂ overvoltage and can be used at current densities > 100 A/cm².

Electrode for Alkali Metal Chloride Electrolysis

SOLVAY S.A. *European Appl.* 707,095A

An electrode consists of an electrically conductive substrate coated with Pt, Ir oxide and Sn oxide. The coating comprises, by wt. %: 12–17 Pt, 30–40 Ir oxide and 53–58 Sn oxide. The anode is used in the electrolysis of an aqueous alkali metal salt, preferably NaCl solution, especially for Cl₂ production. The Ir oxide content gives increased O anion discharge overvoltage and thus improves anodic current efficiency.

Production of Coated Titanium Electrode

NAT. SCI. COUNCIL *Japanese Appl.* 8/20,882

A Ti electrode is plated with an Ir-Pd oxide layer, followed by heating. The Pd oxide provides the electrode with enhanced stability. The plated layer has superior adhesion and the resulting electrode has good electrochemical stability and superior electrochemical catalytic action in acid solutions.

High Oxidation Efficiency Anode

CHLORINE ENGINEERS K.K. *Japanese Appl.* 8/35,089

An electrode film coating applied on a conductive base material comprises 10–45 wt. % Pd oxide, 15–45 wt. % Ru oxide, 10–40 wt. % TiO₂, 10–20 wt. % Pt, and 2–10 wt. % of at least one metal oxide selected from Co, La, Ce and Y. The anode has high oxidation efficiency for hypochlorite preparation, and is formed using a slurry of dispersed Pd and other metal oxides, coated on the electrode, followed by heating in O₂.

Long Life Iridium/Platinum Electrode

TDK CORP. *Japanese Appl.* 8/60,390

An electrode covering layer contains Ir oxide as 2–90 at. % Ir, 2–92 at. % Pt and 3–75 at. % Si on a conductive base material. It may also contain ≤ 30 at. % oxides of Ta, Ti, Nb, Zr and Sn. The electrode has superior durability and is produced by pyrolysis.

Anode for Generating Oxygen

DAISO CO. LTD. *Japanese Appl.* 8/109,490

An anode for O₂ generation during electrolytic processes is produced by roughening the surface of the Ti alloy base by blast treatment, chemically etching the base and forming a 1–5 μm thick Ta (alloy) intermediate layer. Upon this is placed an active coating of a Pt group metal oxide.

ELECTRODEPOSITION AND SURFACE COATINGS

Electroless Plating of Iridium

AGEN. OF IND. SCI. & TECHNOL.

European Appl. 715,000A

An electroless plating bath for Ir plating contains a hydrazine complex of Ir of pH 1–7 and a hydrazine hydrate and/or hydrazinium salt, Ir halide and/or halogenoiridate in molar ratio 1:10 and pH < 3. The bath gives improved Ir adhesion and produces a cation exchange membrane junction for a solid electrolyte H₂O electrolytic cell.

Immersion Plating a Solderable Noble Metal

R.D. CHEM CO.

World Appl. 96/11,751A

A composition for immersion plating a solderable noble metal, especially Pd, onto a substrate which is sufficiently electropositive to reduce the metal in the composition comprises an aqueous solution of a Pd salt and a complexing agent, such as oxalic, glycolic or citric acid, the pH of the solution being 0–5.5. It is used especially for plating Pd onto the Cu (alloy) surface of a PCB for forming contacts, etc. The method is fast, efficient and non-toxic and gives a thin, non-permeable Pd layer of high solderability.

Formation of Aluminide Layers

GENERAL ELECTRIC CO.

U.S. Patent 5,494,704

Aluminide coatings for protecting engine components, such as in turbines in gas, steam and nuclear power generators in harsh environments, comprise a homogeneous biphasic coating 25–1250 μm thick, formed by simultaneous CVD of Al and Pt from Al-alkyl halide precursor and Pt-β-diketonate or dialkyl-Pt-diene, respectively, at ≤ 600°C. Al is deposited in ≥ 10 fold stoichiometric excess to Pt. Pt and Al are interdiffused by heat to form an aluminide layer containing trace amounts of Pt.

Stable Platinum Coating Solution

DOW CHEM. CO.

U.S. Patent 5,503,663

A stable coating solution for electro-active valve metal anodes comprises two soluble Pt group metal compounds and a soluble valve metal compound dissolved in an anhydrous mixture of a lower alcohol(IV) and an anhydrous volatile acid (V). The coating solution is stable for long periods of time and is used to produce dimensionally stable anodes for use in electrolytic cells with permselective membranes.

Palladium-Molybdenum Alloy Plating Bath

TANAKA KIKINZOKU KOGYO K.K.

Japanese Appl. 8/20,887

Pd-Mo alloy plating is performed in a Pd-Mo alloy plating bath of pH 4.0–10, containing 0.3–5 g/l Pd, 2–50 g/l Mo, and 0.1–5.0 mol/l NH₃ or NH₄ salt, as NH₄ ions, at bath temperatures of 30–70°C. The Pd-Mo coatings have high catalytic oxidation ability.

APPARATUS AND TECHNIQUE

Gas Detection Apparatus

TOYOTA JIDOSHA K.K. *European Appl.* 710,835A

A gas detector for detecting CO or organic compounds contained in H₂-containing reactant gas, uses an electrolyte membrane between electrodes carrying Pt/C catalysts. The electrodes are connected by a resistor across which potential is measured. The reactant gas and O₂ are supplied to the separate electrodes via conduits. The electrodes are made of a C cloth weave of C fibres, and C powder with a Pt catalyst is inserted into the pores of the C cloth.

Thick Film Heater

GENERAL MOTORS CORP. *European Appl.* 720,018A

A thick film heater for a combustion engine exhaust sensor, comprises a substrate with a heating element formed on it. The heating element is prepared from two inks, the first contains 50–75 wt.% of Pt, Pd, Rh and/or Ag, and 5–25 wt.% of insulator selected from Al₂O₃, etc., deposited in a serpentine shape; and the second contains 70–85 wt.% of Pt, Pd, Rh and/or Ag and 0–5 wt.% of Al₂O₃, SiO₂, and/or cordierite, deposited in the shape of leads connecting to the serpentine pattern. The sensor has a higher serpentine temperature and lower contact temperature and cost.

Reduction of CO in Vehicle Crash Airbags

E. I. DU PONT DE NEMOURS & CO.

World Appl. 96/10,001A

CO and NO_x in vehicle crash airbags are removed by inclusion of oxidation agents to convert CO to CO₂ and a supported Pd salt in contact with a second oxidation agent. A device positioned within the airbag to reduce CO and NO_x concentrations after inflation is also claimed. The airbag is inflated by an organic pyrotechnic inflator. Devices are small, low cost, easily made and retain their activity for several years.

Dechlorination of Organic Compounds

RESEARCH CORP. TECHNOL. INC.

World Appl. 96/20,137A

Chlorinated organic compounds, such as polychlorinated biphenyls, in groundwater effluent are dechlorinated by contact with a palladised Fe bimetallic system, Pd-Fe, either above-ground or in-ground. An apparatus for dechlorination, involving a H₂O treatment device, is also claimed. Pd-Fe comprises 0.1–20 wt.% Pd deposited on Fe metal, with Fe in the form of wool, mesh, grating, etc. The groundwater is pumped to a filter, portable tank, etc., perforated or non-perforated wall or panel, tube or fluidised bed.

Gas Sensor for Carbon Monoxide

MATSUSHITA DENKI SANGYO K.K.

Japanese Appl. 8/75,691

A CO gas sensor has a fibre portion consisting of SiC fibres coated with Pt, Pd and/or Rh with electrodes at both ends, and a heater portion. The sensor has good response and durability, and can be produced at low cost. CO in combustion exhaust gas or the atmosphere is detected with high sensitivity.

Oxygen Sensor

ROBERT BOSCH G.m.b.H. *German Appl.* 4,436,222

A sensor for measuring O₂ in exhaust gas mixtures from ICE compares results from electrochemical measurements and reference probes separately embedded in a common substrate. The measuring probe has a diffusion barrier and two porous Pt electrodes, one of which is connected to the gas to be measured, and between which an electrolyte of Y₂O₃-stabilised ZrO₂ is fixed. Destruction of the sensor caused by differing expansion coefficients of the different materials is avoided due to the incorporation of both probes in the common substrate.

HETEROGENEOUS CATALYSIS

Exhaust Gas Cleaner

AGENCY OF IND. SCI. & TECHNOL.

European Appl. 710,499A

Exhaust gas cleaner (1) with a NO_x removal catalyst comprises supports of porous inorganic oxides carrying 0.2–15 wt.% Ag and/or its compounds, 0.2–30 wt.% Cu oxide or sulphate, and 0.01–5 wt.% Pt, Pd, Ru, Rh, Ir and/or Au. (1) removes NO_x from gas streams containing NO_x and O₂ in amounts above stoichiometric with respect to unburned components in the stream by contact at 150–600°C in the presence of a hydrocarbon or O₂-containing organic compound as reducing agent. The NO_x stream is from stationary combustion devices, gasoline or diesel engines.

Combustion Catalyst

INST. FRANCAIS DU PETROLE

European Appl. 712,661A

A combustion catalyst comprises a monolithic support; 100–400 g/l porous refractory inorganic oxide support; 0.3–20 wt.% Ce; 0.01–3.5 wt.% Fe; and 3–20 g/l Pd and/or Pt. The catalyst is used for the catalytic combustion of hydrocarbons, CO and/or H₂ in a one-stage process, preferably at < 1100°C.

Catalyst for Dehydrogenation of Paraffins

DEGUSSA A.G.

European Appl. 714,872A

A catalyst for the dehydrogenation of 6–15C paraffins contains an inorganic support, 0.01–5% of a Pt group metal, 0.01–5% of Sn, Ge and/or Pb as promoter, and 0.01–20% of an alkaline earth metal as modifier. The stability of this catalyst is higher than that of catalysts containing alkali metals.

Three-Way Exhaust Gas Purification Catalyst

TOYOTA CHUO KENKYUSHO K.K.

European Appl. 715,879A

The catalyst comprises CeO₂ of 5–100 nm average particle diameter and a noble metal, selected from Pt, Pd and Rh, on a porous support. The average particle diameter is large enough to prevent the oxide entering the pores of the support. A slurry-like solution is coated onto the surface of the support and calcined. Pt group metal is then loaded onto the coating layer. The catalyst has good heat resistance, can operate in an oxidising or reducing atmosphere, and has a very large O₂ storage capacity from CeO₂.

Preparation of High Octane Gasoline

A. PUSCHKAREW

World Appl. 96/7,712A

Gasoline with a high octane number is prepared by catalytically reforming a mixture of H₂ and hydrocarbons by feeding the mixture through a Pt-Re catalyst in a hollow cylinder, at 450–500°C and a pressure of 1.5–3 MPa, vertically to the cylinder axis. The process of feeding the mixture from the axis to the periphery of the cylinder is novel, resulting in improved yield and quality of the products.

Hydrogenation of Lubricant Hydrocarbons

MOBIL OIL CORP.

World Appl. 96/9,359A

A hydrocarbon lubricant with bromine number > 5 is hydrogenated to bromine number < 3 over a Pt group metal catalyst, preferably Pt, Pd, Ir or Re, supported on an inorganic, porous, crystalline material, such as kieselguhr, Al₂O₃, SiO₂, etc. The support pores have diameter ≥ 13 Å and at least one XRD peak with *d*-spacing > 18 Å and relative intensity of 100 after calcination. The catalyst can give very low bromine number products under mild conditions.

Preparation of Nitrobenzene Derivatives

RHONE-POULENC RORER S.A. *World Appl.* 96/10,010A

The preparation of nitrobenzene derivatives substituted in position 2 by a carboxylic acid-containing group comprises dehalogenation of the corresponding halogenated compounds (1) using triethylammonium formate in the presence of a Pd/C catalyst. The catalyst contains 0.002–0.1 moles Pd/C per mole of (1), in acetonitrile or THF, at between 50°C and the boiling point of the mixture. This non-polluting, industrial-scale process gives the required compounds in good yields.

Production of Mono-olefin

UNIV. MINNESOTA

World Appl. 96/13,475A

The production of a mono-olefin from a gaseous paraffinic 2+ C-hydrocarbon comprises reacting the hydrocarbons and molecular O in the presence of a Pt catalyst consisting of Pt supported on a ceramic foam monolith of oxides of Al, Zr, Ca, Mg, Hf and/or Ti. The catalyst has high selectivity and activity with less coking. High yields of mono-olefins are obtained.

Hydrogenation Catalyst

IMPERIAL CHEM. IND. PLC *World Appl.* 96/17,683A

A hydrogenation catalyst comprises Pd and Pt in a wt. ratio of 2:1 to 500:1 on a C support. It is used at elevated temperatures for hydrogenation of (hydro) halofluorocarbon for the production of hydrofluorocarbon, useful as solvents, refrigerants, etc. The catalyst has improved activity and selectivity.

Isomerisation of Linear Paraffin Feedstock

SHELL CANADA LTD.

World Appl. 96/18,705A

The isomerisation of a hydrocarbonaceous 5+ C linear paraffin feedstock with a gasoline range boiling point involves contacting the feedstock H₂ at elevated pressures in the presence of a metallic Pt and Pd catalyst on an acidic Al₂O₃ or molecular sieve supports to produce branched paraffins. The catalyst has high activity, selectivity and stability, and yields upgraded paraffins.

Conversion of Methane

EXXON RES. & ENG. CO.

U.S. Patent 5,504,262

A mixture of MeOH and EtOH was produced by contacting CH₄, H₂O and an acidic aqueous solution of an electron acceptor selected from Fe₂(SO₄)₃ and Fe(ClO₄)₃, of pH < 3 with a Pt group metal catalyst selected from Pd and Pt having a particle diameter of ≥ 100 Å at ≥ 60°C. CH₄ gas is primarily converted into EtOH with smaller amounts of MeOH being produced. The process offers high thermodynamic efficiency and low capital cost.

Reforming Hydrocarbon Feedstock

UOP

U.S. Patent 5,507,939

Catalytic reforming of hydrocarbon feedstock, especially gasoline range hydrocarbons, involves contacting a feed of hydrocarbons and H₂ in a first reforming step over Pt/Al₂O₃ catalyst at 1–20 atm and 260–560°C to convert S compounds to H₂S and produce a first effluent, which is contacted with a second reforming Pt group metal catalyst to remove H₂S at 1–20 atm and 425–560°C. The second effluent, in the presence of H₂, undergoes dehydrocyclisation with a Pt group metal catalyst on a non-acidic L zeolite. A halogen-free aromatic-rich effluent is produced. The gasoline products have improved octane number. The catalyst has good S tolerance.

HOMOGENEOUS CATALYSIS

Production of 2-Methyl-1,4-Naphthoquinone

KURARAY CO. LTD.

European Appl. 704,421A

Production of 2-methyl-1,4-naphthoquinone (1) comprises reacting H₂O₂ and/or an organic peracid with 2-methylnaphthalene in a solvent containing a carboxylic acid, in the presence of a Pd compound and 5–100 g H₂SO₄/l of carboxylic acid. The process allows easier heat removal and (1) is menadione or vitamin K₃, and has antithaemorrhagic activity.

Preparation of 1-Arylalkynes

HOECHST A.G.

European Appl. 709,357A

1-Arylalkynes are prepared in high yield by cross-coupling terminal alkynes with aromatic halogen or perfluoroalkylsulphonate compounds catalysed by Pd in the presence of H₂O-soluble complex ligands. The reaction medium forms an aqueous and an organic phase and the Pd is added as a compound soluble in the organic phase or in solid form as metal. Arylalkynes are useful as liquid crystal compounds, etc. The process gives good yields with very high purity of 1-arylalkynes. The reaction is very chemoselective.

Preparation of Modified Purine

NEXSTAR PHARM. INC.

World Appl. 96/16,972A

Modified purines are prepared by reaction of a purine compound containing a leaving group attached to a C atom with an organotin compound in the presence of a PdL₃ catalyst, where L is a ligand, such as PPh₃, (*o*-tol)₃P, CH₃CN, DMSO, etc. The PdL₃ catalyst accelerates the formation of products. The purines can be used in oligonucleotides to interact with biological targets and may be useful in diagnostics.

Ene-yne Unsaturated Accelerators

DOW CORNING CORP. *U.S. Patent 5,493,045*

A hydrosilation process involves contacting a Si hydride of formula $R_1H_bSiX_{4-a-b}$, where $R_1 = 1-20C$ alkyl; 4-12C cycloalkyl or aryl; X = halogen or -OR1; $a = 0-3$; $b = 1-3$; and $a+b = 1-4$; and/or an unsaturated reactant and/or a Si compound, both comprising optionally substituted unsaturated organic compounds, with a Pt compound or complex catalyst, and an ene-yne unsaturated compound accelerator. The accelerator improves the yield.

CHEMICAL TECHNOLOGY

Hydrogen Cyanide Preparation

E.I. DU PONT DE NEMOURS & CO.

U.S. Patent 5,529,669

Hydrogen cyanide is produced by passing a mixture of NH_3 vapour and a hydrocarbon gas at a pressure, at least equal to atmospheric pressure, over a catalyst containing 0.1-20 wt.% Pt subjected to microwave heating, to reach the reaction temperature. HCN is then recovered. HCN can be produced efficiently on a small scale with minimal energy wastage.

ELECTRICAL AND ELECTRONIC ENGINEERING

Magneto-Optic Thin Film for Optical Disk

M. TAKAHASHI

World Appl. 96/8,008A

A magneto-optic film is composed of an alloy $Mn_xSb_yPt_z$, where x, y and z are proportions in atomic per cent: $45 \leq x \leq 56$; $40 \leq y \leq 48$; and $2 \leq z \leq 15$; $x + y + z = 100$. The alloy has a specific diffraction intensity ratio. The film thickness is 10-300 nm and is produced by heat treating the substrate at $> 100^\circ C$ in vacuum. The resulting films have high Kerr rotation angle in the short wave band.

Formation of Ultra-Pure Platinum Film

MITSUBISHI MATERIALS CORP.

Japanese Appl. 8/20,870

Ultra-pure Pt film, for contact or wiring a semiconductor device, is formed by decomposing the vapour of an organic Pt compound accumulated on a substrate by irradiation with light of wavelength ≥ 240 nm. The light breaks the bonding between Pt and a ligand at low temperatures, without decomposing the ligand. Pt is deposited at a high rate and forms the film at $\leq 150^\circ C$ by photodecomposition MOCVD.

Metal Thin Film Magnetic Recording Medium

KUBOTA CORP.

Japanese Appl. 8/31,638

A magnetic thin recording medium consists of a substrate on which three layers are joined sequentially: a foundation, a magnetic and a protection layer. The magnetic layer comprises 6-20% Cr and 0.5-7% Cu, with the remaining per cent of the composition being a Co alloy: 72% Co-20% Pt-8% B. The crystal grain is miniaturised. The coercive force of the medium is high and the noise ratio is reduced. Magnetic separation is promoted.

Magnetic Recording Medium

DENKI KAGAKU KOGYO K.K. *Japanese Appl. 8/31,639*

The recording medium for a vertical recording system, such as the hard disk of a computer, has an interface layer positioned between a soft magnetic layer and a magnetic recording layer made of Pt and/or Pd. The recording type magnetic layer comprises Co-Cr system alloy and Co-Cr-Ta-Pt system alloys, and is positioned on the interface layer. The layers are covered by a protective layer. The recording medium increases the coercive force, improves recording and reproducing characteristics, and the recording density.

Fine Palladium Alloy Wire

MITSUBISHI MATERIALS CORP.

Japanese Appl. 8/102,472

A fine Pd alloy wire for a semiconductor device, comprises 0.2-100 ppm rare earth element, 1-5000 ppm Ag, 0.2-100 ppm Ca, 0.2-100 ppm of one of Be, Ge, Si, In or Ir, and Pd, and impurities. The wire is used as a bonding wire for semiconductor devices and provides highly reliable bonding.

Ceramic PWB for Electronic Device

MATSUSHITA ELECTRIC WORKS LTD.

Japanese Appls. 8/102,578-580

A PWB for electronic devices, such as portable telephones, tape recorders, etc., has a ceramic substrate on which a metal layer of Ag and Pd is formed between the substrate and a Cu layer. A solder consisting of a metal paste of Pt and Pd is applied to the surface of the ceramic substrate and baked at $700-850^\circ C$ in air. The Cu layer conceals the first metal layer. The ceramic PWB prevents reduction of contact in the electric conduction circuit due to solder component diffusion, and improves die and wire bonding.

Magnetron Sputtering Target

LEYBOLD MATERIALS G.M.B.H.

German Appl. 4,438,202

A magnetron sputtering target, consisting of a Co alloy with 6-20 at.% (exclusive) Pt and 10-22 at.% (exclusive) Cr, has a mainly cubic phase structure and a magnetisation curve $M(H)$ with an inversion point $H = 0-5$ kA/m, with $dM/dH (H=0) < dM/dH$ (inversion point); the initial slope at $H=0$ is ≤ 30 . The target has an extremely large scatter field and very low magnetisation in low H field strength regions.

MEDICAL USES

Ruthenium Complexes for Immunosuppressants

PROCEPT INC.

U.S. Patent 5,512,687

Ru complexes for preventing transplant rejection have formula RuM_mZ_n (1), where the Ru oxidation state = +2 or +3; M = a 5-membered heterocyclic amine; $m = 6$; Z = a charged counter-ion, rendering the overall charge neutral; and $n = 0-4$. (1) can be used as drugs for T lymphocyte mediated auto-immune diseases.

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