NEW PATENTS

METALS AND ALLOYS

Magnetic Alloy

KOMAG INC. *European Appl.* 669,610A

A magnetic alloy with good magnetic properties and excellent corrosion resistance comprises, in at.%: ≤ 20 Pt, ≤ 10 Ni, ≥ 75 Co, ≤ 10 Ta and ≤ 10 Ti. This is incorporated in recording media consisting, in order: a substrate, a plated layer, a nucleation layer, a magnetic alloy layer, a protective overlayer and a top lubricant layer. The media are used in rigid disk drives for computer data storage.

Manufacture of Silver-Palladium Alloys

E.I. DU PONT DE NEMOURS & CO. *U.S. Patent* 5,429,657

Finely divided particles of Ag-Pd alloy are manufactured by aerosol decomposition of an aqueous solution containing nitrates of Ag and Pd at a temperature below the melting point of the alloy but above the decomposition temperature of the nitrates. The powders are not hollow, are of high purity and are spherical. The alloys are used in electronics and dentistry, in particular in electrode materials for multilayer ceramic capacitors and in printed circuits.

Silver-Palladium Powder Manufacture

DAIDO TOKUSHUKO K.K. *Japanese Appl.* 7/150,206

A Ag-Pd powder is made by regulating the pH of a Ag-Pd solution in HNO_3 acid by adding a base, an aromatic reducing agent as the first reducing process, and a non-aromatic reducing agent as the second reducing process. The powders are used for electroconductive paste electrodes for hybrid ICs, multilayer ceramic capacitors and chip resistors.

ELECTROCHEMISTRY

Hydrogen Peroxide Production

KEMIRA CHEM. O.Y. *European Appl.* 672,617A

H_2O_2 is produced by the anthraquinone process by feeding H_2 or an H_2-containing gas into the top part of a fixed bed reactor, flowing the H_2 downwards to hydrogenate the anthraquinone derivative over a stationary catalyst of supported Pd catalyst mesh, and removing the hydrogenated working solution from the reactor. The process is highly efficient, with good yields; catalyst regeneration problems are much reduced and a steady supply flow into the reactor is achieved. A dense, almost foam-like dispersion is obtained in the catalyst bed.

Iridium Electrode Base Material

NIPPON STEEL CORP. *Japanese Appl.* 7/166,350

Ir is ion-implanted onto the surface of a base material which has a Ti metal layer on Ti metal. The Ir oxide coated electrode has high durability and no flaking occurs, even under heavy usage. The electrode is used as an insoluble electroplating electrode.

Electrolytically Ionised Water

KOBE STEEL LTD. *Japanese Appl.* 7/171,571

An electrode for the electrolytic ionisation of H_2O consists of Ta or Nb base material coated with at least 50 vol.% of Pt, IrO_2, PtO or Rh_2O_3. The electrolysis is carried out by controlling the anode potential or the voltage between the anode and cathode. The electrode can produce electrolytic ion H_2O, alkaline H_2O from a cathode room and acidic H_2O of pH 1–2 from an anode room. It has a longer operating life so that production is continuous over a long time.

ELECTRODEPOSITION AND SURFACE COATINGS

Palladium Alloy Plating Solution

S. S. MOON *British Appl.* 2,287,717A

A Pd alloy plating composition comprises (in g/l): 4–20 Pd ion, 0.3–2 Au ion, 5–100 conductive salt and 0.5–20 complexing agent. The Pd source is preferably a Pd(II) diammonium, diamine, diammonium or tetramine dichloride, diamine dinitride, oxide, nitrate or cyanide. The composition may also contain 0.3–5 g/l Ni, Co, Cu, Sn, Se, W, Mo and/or Ti ions as alloying metal. The plated metal has excellent stability, solderability and flexibility. Pd-Au alloy can be electroplated onto lead frames, PCBs and connectors.

Lustrous Noble-Metal Decorations

CERDEC A.G. *European Appl.* 668,265A

Mono-noble metal dithiolates contain a noble metal of Pt(II), Pd(II), Rh(III), Au(I) or Ag(I); a tetravalent 2–10C organic group; an hydrophilic group, and a mineral acid or carboxylic acid anion. Decals based on these compounds provide highly lustrous noble metal decoration. The decorated substrates of glass, porcelain or ceramic can be baked. The decoration is non-porous, stain-free and has good adhesion.

Protective Coating for Lead Frames

NAT. SEMICONDUCTOR CORP. *World Appl.* 95/18,464

A protective coating comprises, in sequence, layers of Ni, Cu, Ag and Pd. Also claimed is the method for forming the coating by electrolytic deposition. Preferably the layers are pure metal or alloys of thickness (in μm): 1.25–2.5 Ni, 0.08–0.38 Cu, 0.13–1.0 Ag and 0.08–0.63 Pd. The finish is bondable, solderable, free of lead and corrosion resistant.

Plating of Precious Metals

TANAKA KIKINZOKU KOGYO K.K. *Japanese Appl.* 7/157,879

Plating of Pt, Pd, Rh, Ir, Os, Au, Ag, Cu, Pb, Ni and Co on a matrix surface is achieved by allowing H_2 to occlude into the matrix by circulation or by pressure. The matrix is then dipped into the metal-containing aqueous solution and metal is deposited onto its surface by reduction.

Platinum-Coated Layer Formation

TANAKA KIKINZOKU KOGYO K.K.

Japanese Appl. 7/188,934

Pt-coated layers are formed by mixing aqueous chloroplatinic acid solution and propargyl alcohol, coating onto a base material and baking. Grains of Pt metal are heat-decomposed, deposited and baked onto the base at 200–250°C, which is a relatively low temperature. Such a Pt-coated layer has ~ 1.6 times the specific surface of a usual Pt-coated layer. The method is used to form a Pt-coated layer for an insoluble electrode, etc., used in electrochemical reactions.

Anti-Allergic Noble Metal-Coating

SEIKO INSTR. INC. *Japanese Appl.* 7/188,945

A noble metal coated ornament comprises a Cu-Sn alloy coating containing 30–60% Sn as the primary layer at 0.5 μm thickness and a top coating of Pd, not containing Ni, at a thickness of 0.1–5 μm. As an example, a wrist watch casing made of bronze was coated with 3 μm of Cu-Sn alloy and then coated with Pd to a thickness of 3 μm. The coating is anti-allergic.

APPARATUS AND TECHNIQUE

Silicone Coated Base Material

SHINETSU CHEM. CO. LTD. *European Appl.* 669,419A

A flame resistant, silicone coated base material for automotive air bags comprises an organopolysiloxane, an organohydrogen polysiloxane, an organic Si compound containing an epoxy or a trialkoxysilyl group metal oxide powder and catalyst. The catalyst is Pt on a C, NiO_2, FeO, FeO_2, Fe_2O_3, CoO_2, CeO_2 or TiO_2 support. The air bag base material is formed on a fibrous base material, such as polyamide fibres, polyester fibres and their woven or non-woven fabrics.

Oxygen Concentration Detecting Element

UNISIA JECS CORP. *Japanese Appl.* 7/128,276

An oxygen concentration detecting element consists of a ZrO_2 tube having an inner and an outer electrode, with a vapour deposited Pt layer on the outside surface of the tube, which covers the outer electrode. A sintering-prevention agent is adhered to the Pt layer. Deterioration of the catalytic activity of the Pt catalyst layer is prevented during prolonged use.

Treatment Process for Oxidant

NIPPON SHOKUBAI CO. LTD. *Japanese Appl.* 7/214,078

An oxidant in sea- and fresh-water is treated by contact with a Pt/TiO_2-ZrO_2 catalyst. The oxidant is decomposed by contact with the solid catalyser.

Garnet Crystal

NAMIKI SEIMITSU HOSEKI K.K.

Japanese Appl. 7/215,799

A garnet crystal for a magnetic-bubble or magneto-optical device, is grown using a liquid-phase epitaxial device, which has a Pt-based crucible, a substrate binder and a non-magnetic garnet substrate, which has a crystal growing face. No cracks are formed and reproducibility is good.

HETEROGENEOUS CATALYSIS

Hydrogenation of Aromatic Hydrocarbons

MITSUBISHI CHEM. CORP. *European Appl.* 659,718A

A cycloolefin is produced by partial hydrogenation of a monocyclic aromatic hydrocarbon in the presence of H_2O and a Ru/SiO_2 catalyst, modified by Zr oxide to improve Ru dispersion and catalyst life, optionally in the presence of a metal salt. The catalyst has good selectivity for cycloolefin, ~ 60–80% at ~ 30% conversion. Cycloolefins are important intermediates for lactams and dicarboxylic acids, etc.

Exhaust Gas Cleaner

RIKEN CORP. *European Appl.* 661,089A

An exhaust gas cleaner, for removing NOx by reduction and removing unburned CO and hydrocarbon components by oxidation from exhaust gas containing NOx and O_2 in an amount above stoichiometric is claimed. It comprises a first catalyst of 0.2–15 wt.% Ag/porous inorganic oxide support, optionally in combination with ≤ 1 wt.% Pt, Pd, Ru, Rh, Ir and/or Au, or ≤ 5 wt.% W, V, Mn, Mo, Nb and/or Ta; and a second catalyst of a porous inorganic oxide supporting ≤ 5 wt.% Pt, Pd, Ru, Rh, Ir and/or Au and optionally ≤ 10% W, V, Mn, Mo, Nb and/or Ta. The cleaner is placed in the flow path of the exhaust gas. Reducing agents are added to the upstream side of the cleaner.

Hydrogenation of Carboxylic Acids

BP CHEM. LTD. *European Appl.* 662,343A

A catalyst for hydrogenation of carboxylic acids to alcohols or esters comprises an alloy of at least one Group VIII metal, such as Pd, Rh and Ru, and at least one other metal, such as Ag, Au and Cu, on a support of high surface area graphitised (HSAG) C, graphite or activated C. A preferred catalyst comprises an alloy of Pd-Ag and Re supported on HSAG C.

Three-Way Exhaust Gas Catalyst

E. G. PAPADAKIS et al *European Appl.* 665,047A

A three-way catalyst, for treating car exhaust gas, industrial and power station waste gases, involves oxidation of CO and light hydrocarbons and NOx reduction, and comprises dispersed Rh, Pd and Pt each on a different monolithic honeycomb-type support. The metal/support pairs are macroscopically uniform catalyst in the form of pellets or are applied as a washcoat in three layers on the monolithic substrate.

Catalyst for Treatment of ICE Exhaust Gases

INST. FRANCAIS DU PETROLE

European Appl. 665,048A

A catalyst for exhaust gas treatment comprises a support with a porous refractory inorganic oxide layer and an active phase of: 0.3–4.4 % Ce, 0.1–3.5 % Fe, 0.003–0.04 % Pt and balance of at least one refractory inorganic oxide. The formation of sulphates and sulphites upon oxidation of SO_2 is prevented. The catalyst has good thermal stability and resistance to ageing and is used to treat the exhaust gases from compression ignition (diesel) engines, especially for commercial vehicles with a total weight of ≥ 3.4 tonnes.

Exhaust Gas Purification Catalyst

CATALER IND. CO. LTD. *European Appl.* 666,103A

An exhaust gas purification catalyst comprises a porous support loaded with a NOx storage component of alkali, alkaline earth and/or rare earth element; and an adjacent catalyst of Pt, Pd and/or Rh; and CeO_2 positioned away from the Pt group metal. The support is Al_2O_3, zeolite, SiO_2, ZnO_2 and/or SiO_2/Al_2O_3. Oxidation of hydrocarbons and CO is controlled by the release rate of O_2 from CeO_2, thus giving enhanced NOx reduction in fuel-rich atmospheres.

Preparation of Hydroxy-Benzaldehyde

RHONE POULENC CHIM. *European Appl.* 667,331A

A hydroxy-benzaldehyde is produced by oxidation of the corresponding hydroxy-benzylic alcohol using O_2 in an aqueous medium containing an alkaline agent, in the presence of a Pt-based catalyst, such as Pt/C, and of a B and a Bi derivative as co-catalyst. The process is particularly applicable to the preparation of salicyl-aldehyde from saligenol. The joint presence of B and Bi derivatives, with the Pt-based catalyst, gives greatly increased reaction yields. The final product is obtained without isolating the intermediates.

Production of Fluoroaniline Compounds

HOECHST A.G. *European Appl.* 667,337A

Fluoroanilines (1) are produced by the reaction of (chloro- or bromo)fluoronitrobenzene with H_2 in the presence of a Pd catalyst, such as 5 wt.% Pd/C, and a H_2O-insoluble amine which forms a H_2O-insoluble hydrohalide, and optionally, in an inert solvent. The method produces (1) in good to very good yields and purity; (1) are plant protectants and pharmaceutical intermediates. The process can be operated on a plant scale, being readily reproducible, non-corrosive and not producing environmentally harmful by-products.

Catalyst Support for Fluidised Bed

STANDARD OIL CO. OHIO. *European Appl.* 672,453A

Supports for the manufacture of vinyl acetate catalysts comprise a mixture of inert, microspherical SiO_2 particles of pore volume 0.2–0.7 cm^3/g, of surface area 100–200 m^2/g with ≥ 50 % of the particles being < 100 μm. The support is impregnated with Pd and Au salts and alkali metal salts. Vinyl acetate is produced by this catalyst by reaction of ethylene, acetic acid and O_2 in a fluidised bed. Continuous addition of the catalyst maintains peak performance and almost eliminates the need for catalyst changes.

Exhaust Gas Purification Catalyst

KEMIRA O.Y. *World Appl.* 95/17,249A

A catalyst for the purification of gasoline engine exhaust gases comprises a catalyst body with numerous through-flow channels for exhaust gas. The catalyst body material is coated with Al_2O_3 slurry and La oxide or soluble salt, and with Pd. The support additionally comprises oxides or soluble salts of Eu and/or Sm. The Pd content is 2–15 g/l of catalyst honeycomb. Also claimed is the catalyst preparation. The catalyst has a maximum uniform surface and gives improved conversion of CO, hydrocarbon and NOx.

Carbon Monoxide and Hydrogen Preparation

SHELL CANADA LTD. *World Appl.* 95/18,063A

CO and/or H_2 are prepared from gaseous hydrocarbon feedstock, such as CH_4 or natural gas, by partial oxidation of the feedstock using an O_2-containing gas, which also contains N, by a catalyst containing Rh, Ir or Pt. At least part of the products is subjected to a second stage process to remove NH_3 and/or HCN. Only small amounts of HCN and/or NH_3 are produced by this method and are thus easily removed at the second stage.

Production of Propylene

DOW CHEM. CO. *U.S. Patent* 5,430,215

Hydrodechlorination of 1,2,3-trichloropropane (TCP) to produce propylene, in preference to propane, in ≥ 10% yield, comprises reacting TCP and H_2 in the presence of a supported catalyst consisting of Pt in elemental or compound form. Propylene is produced in ≥ 20% yield, such as 42% yield with 47% selectivity. The process allows extension of the C value, which would otherwise be lost during incineration of TCP. The propylene can be recycled to produce extra allyl chloride for the epichlorohydrin process.

Dehydration of Lower Alkanes

PHILLIPS PETROLEUM CO. *U.S. Patent* 5,430,220

A process for dehydrogenating a 2–8C alkane to an alkene, in the presence of steam and a catalyst of Pt, Sn and a Zn aluminate-containing support, is improved by impregnating the support with a solution formed by combining aqueous solutions of a Pt compound and oxalic acid, and a Sn compound and an inorganic acid. This method of catalyst preparation increases activity and selectivity (especially to monoolefins), and decreases the rate of deactivation.

Catalyst for Synthesis of Chlorine Dioxide

DEGUSSA CORP. *U.S. Patent* 5,435,984

ClO_2 is generated by contacting a precursor in an aqueous medium with a catalyst consisting of a support comprising α-Al_2O_3, γ-Al_2O_3 or SiO_2, and containing La_2O_3 and/or Nd_2O_3, which is impregnated on the outer surface with Pd, or Pd and another Pt group metal, or Pd and a Group IB metal. Preferably, the reaction temperature is 5–80°C, pH is 1–8, and contact time between catalyst and precursor is 0.01–20 s. The rate of deactivation of the catalyst is slower than for prior catalysts.

Generation of Carbon Monoxide and Hydrogen

PRAXAIR TECHNOLOGY INC. *U.S. Patent* 5,441,581

CO and H_2 atmospheres for heat treating ferrous and non-ferrous metals, alloys and ceramic and/or metal powders are obtained by an endothermic generation process. Input gases are passed through a conduit and onto a catalyst comprising a noble metal, such as Pt and particularly Rh, loaded on a porous ceramic carrier, at 750–900°C. Output gases, of CO and H_2, with space velocity of ≥ 10,000 units/h are produced. The reactor operates autothermally at very high space velocities and provides a buffered atmosphere to allow the introduction of N_2 into the reducing furnaces.

Hydrogenation of Acetylenic Glycols

AIR PROD. & CHEM. INC. *U.S. Patent* 5,444,170

The hydrogenation of acetylenic compounds obtained by condensation of ketones or aldehydes with acetylene, comprises reaction with H_2 under suitable conditions to give paraffins, and uses a co-catalyst system containing Pd and Pt. The method completely hydrogenates the acetylene compounds, which are contaminated with Pd catalyst poisons. The system gives excellent rates of hydrogenation and remains usable for a long time without deactivation.

Hydrodehalogenation Catalyst

E. I. DU PONT DE NEMOURS & CO. *U.S. Patent* 5,447,896

A hydrodehalogenation catalyst is prepared by impregnating a C support of ash content < 0.2 wt.% with a combination of metals consisting of 5–95 wt.% Au and 95.5 wt.% of at least one metal selected from Ru, Rh, Pd, Os, Ir and Pt, at ~ 350°C. The impregnated C is dried in air at 130–150°C, and heated in a reducing atmosphere at ~ 350°C to provide metal in a reduced state. The addition of Au prevents sintering and increases the durability and mechanical strength of the catalyst.

Preparation of Tercyclohexane from Benzene

MOBIL OIL CORP. *U.S. Patent* 5,449,847

The preparation of tercyclohexane involves passing H_2 and benzene under sufficient conversion conditions over a catalyst comprising an acidic solid of a Group IVB metal oxide, such as Zn; modified with an oxyanion of Group VIB metal, such as W; and an hydrogenation component of Group VIII metals, such as Pt, Pd, Ir, Rh, Os and Ru, preferably Pt. The catalyst has high activity and selectivity for the conversion of benzene to tercyclohexanes.

Removal of Oxygen-Containing Compounds

AGENCY OF IND. SCI. & TECHNOL. *Japanese Appl.* 7/136,461

A gas mixture containing O_2-bearing compounds, such as methanol, aromatic compounds and O_2, is contacted with a Pt group metal catalyst, such as Pt, on a ZSM-5, mordenite or Y-type zeolite support of regular structural micropores. The O_2-bearing compounds can be decomposed in exhaust gas which contains aromatic compounds to suppress their oxidation. Methanol can thus be eliminated from the exhaust gas of a methanol/gasoline engine.

Oxidising Catalyser

BABCOCK-HITACHI K.K. *Japanese Appl.* 7/136,513

An oxidising catalyser for treatment of exhaust gas comprises a base material carrier, such as Al_2O_3, which contains at least one of Ba, Sr, La and Ce; Pt group metal grains, such as Pt or Pd; and a covering TiO_2 layer. The carrier is made of honeycomb or lattice shaped ceramic or metal, ceramic or metal foam or metal lath. The catalyser is used for combustion of inflammable components, and has high resistance to heat and poisoning.

Exhaust Gas Purifying Catalyser

NISSAN MOTOR CO. LTD. *Japanese Appl.* 7/136,518

An exhaust gas purifying catalyser comprises Pd and a perovskite type combined oxide containing one of Co, Mn and one of Fe, Ni, Cu, V or Cr. A catalyser was obtained by adding $Pd(NO_3)_2$ to the perovskite, and $Pd(NO_3)_2$ to γ-Al_2O_3, followed by mixing, drying and burning at 400°C. The Pd catalyser has good NOx purifying ability for exhaust gas from the rich side composition of the theoretical air-fuel ratio, and after high temperature usage for a long period.

Catalyser for Purifying Exhaust Gas

NISSAN MOTOR CO. LTD. *Japanese Appl.* 7/136,524

A catalyser consists of two catalyser layers: an inorganic layer of activated Al_2O_3 containing Pt, Pd and Rh, such as Pt-dinitrodiamine on a honeycomb carrier, and a co-existing layer of Pt and S on a porous carrier. The catalyser is used for purifying exhaust gas in ICE and retains good purifying capacity after exposure to high temperatures from the stoichiometric to the lean range.

Purification Apparatus for Exhaust Gas

MATSUDA K.K. *Japanese Appl.* 7/144,119

The apparatus is composed of tri-dimensional catalyst, such as Pt-Rh/Al_2O_3, at the upper stream side; HC adsorbent, such as zeolite, mordenite, ferrierite and chabazite; and a tri-dimensional catalyst, such as Pd/Al_2O_3, at the downstream side. The activation temperature for the upper stream side catalyst is higher than for the downstream side catalyst, but the activation conditions for both catalysts are almost identical. The purification ratio achieved for HC was 59%.

Material for Purifying Exhaust Gas

RIKEN CORP. *Japanese Appls.* 7/148,435 and 7/148,437

The purifying material is composed of 2 or 3 catalysts. The first catalyst contains Ag or Ag oxide, and at least one of Pt, Pd, Rh, Ru, Ir or Au, on a porous inorganic oxide support, preferably Al_2O_3. The second catalyst comprises at least one of Pt, Pd, Ru, Rh, Ir or Au, on porous inorganic oxide or W and/or V on porous inorganic oxide. The additional third catalyst contains at least one of Pt, Pd, Rh, Ir or Au on a porous inorganic oxide. The material is used for removing NOx effectively, from excess O_2 combustion exhaust gas, over wide temperatures. Residual and non-reacting CO and hydrocarbons are also removed.

Cleaning Exhaust Gas of Lean Burn Engines

NE CHEMCAT K.K. *Japanese Appl.* 7/155,555

Exhaust gas from lean burn engines is contacted with a crystalline Ir silicate catalyst on a carrier of metal carbide and metal nitride, and then with a Pt catalyst made by depositing Pt on a porous carrier by a non-electrolytic plating technique. The catalyst is effective for decomposing NOx in exhaust gas, at 200–500°C, using HC as a reducing agent, and prevents the production of N_2O at 200–300°C. It is stable even in the presence of water vapour.

Improved Production of 1,4-Butanediol

MITSUBISHI KASEI CORP. *Japanese Appl.* 7/165,644

The production of 1,4-butanediol (BDO) and/or tetrahydrofuran (THF) by catalytic hydrogenation of maleic anhydride, maleic acid, succinic anhydride, etc., involves using a catalyst which comprises components from Pt, Ru, and Rh and Sn supported on a carrier. BDO and/or THF can be produced in high yields under comparatively mild reaction conditions.

Glyoxylic Acid Preparation

MITSUI TOATSU CHEM. INC. *Japanese Appl.* 7/173,099

Preparation of glyoxylic acid comprises oxidation of an aqueous solution of glyoxal with O_2 in the presence of Pt and Pd, Pb, Sn, Bi and/or their compounds as a catalyst. The conversion rate of glyoxal is high at 85–97% and the selectivity of glyoxylic acid is 81–82%, compared to 30–71% by other methods.

Preparation of Diarylamine Derivatives

NEW JAPAN CHEM. CO. LTD. *Japanese Appl.* 7/188,131

Preparation of diarylamines comprises dehydrogenation of N-containing compounds in the presence of a metal catalyst chosen from Pt, Pd, Ru, Cu, Cu-Cr and their oxides. Symmetric and asymmetric diarylamines can be produced in high yield under moderate conditions.

Preparation of Amino Alcohols

BASF A.G. *German Appl.* 4,400,591

The preparation of amino alcohols comprises reacting hydroxycarbonyl compounds with H_2 and NH_3 or a primary or secondary amine at 0–300°C and 1–400 bar in the presence of a catalyst of active mass 50–100 wt.% Ru. The process is a one-step synthesis, directly from the hydroxycarbonyl compounds, and gives amino alcohols in high yields and purity. The amino alcohols have use as intermediates, etc.

Ruthenium Catalysts

BAYER A.G. *German Appl.* 4,404,220

Ru catalysts, which are used in the selective preparation of cycloaliphatic polyamine (1) by hydrogenation of aromatic polyamine, additionally contain hydroxides and/or hydrated oxides of Ce and Mn and/or their dehydration products. The catalysts contain 0.1–10 wt.% Ru, and Mn:Ce in weight ratio 0.3–5.1 (as elements), and may additionally contain additives, auxiliaries and/or carriers. The catalyst has high selectivity and the reaction to (1) takes a short time at a high catalyst loading. The catalyst is easily prepared by using readily filtered precipitates.

Tertiary Amino Production

BASF A.G. *German Appl.* 4,407,466

Tertiary amines (1) are produced by reacting nitriles with secondary amines and H_2 at 50–250°C and 5–350 bar in the presence of Pd/oxide support catalyst. (1) are useful as epoxy resin hardeners, polyurethane catalysts, and intermediates for quaternary ammonium compounds, plasticisers, corrosion inhibitors, textile auxiliaries, dye, emulsifiers, etc. The process gives 83–93% yields in continuous operation.

HOMOGENEOUS CATALYSIS

Bicyclic Ketoester Derivatives

MERCK & CO. INC. *World Appl.* 95/19,979A

A bicyclic ketoester derivative is prepared by cyclising a diazo compound in the presence of a Rh catalyst and a catalytic amount of a Lewis acid, which is effective for reducing the formation of the 1-α methyl isomer. The process gives < 1% of 1-α methyl isomer, so requires no separation of the α- and β-isomers.

Arylacetic Acid Ester Derivatives

CIBA GEIGY A.G. *World Appl.* 95/20,569A

Arylacetic acid ester compounds (1) are prepared by reacting boronic acid compounds with methoxy imino-acetic ester, in the presence of a catalyst, such as $Pt(OAc)_2$ and tetrakis(triphenylphosphine)Pd, etc. Compounds (1) are useful as microbiocides, and can be prepared by this method simply and in high yield.

Silicon-Containing Pentacyclic Compounds

AGENCY OF IND. SCI. & TECHNOL. *U.S. Patent* 5,449,800

A new Si-containing pentacyclic compound is prepared by reacting a 1,2-bis(hydrosilyl)benzene containing a monovalent group with a cyclic diene in the presence of a Pt compound. It is used as a raw material for making Si-containing ladder polymers, for heat- and burn-resistant, electrically conductive and non-linear optical materials, under mild conditions.

FUEL CELLS

Perovskite Electrode

FORSHUNGSZENTRUM JUELICH G.m.b.H. *World Appl.* 95/19,053A

An electrode, used as a cathode in a high temperature fuel cell, has perovskite material doped with Pt metal, such as a Pt metal additive based on La ferrite, so as to reduce the reactivity between the electrode and the solid electrolyte of the fuel cell. The electrode is based on ZrO_2 stabilised with Y_2O_3 and is doped with Ir or Ru oxide at 10–1000 ppm. The solid electrolyte fuel cell has improved electrochemical characteristics.

Direct Energy Conversion Device

UNIV. CALIFORNIA *World Appl.* 95/20,246A

A direct energy conversion device, especially for a H_3PO_4 fuel cell, has a pair of C aerogel electrodes (1) loaded with a Pt metal catalyst, such as Pt, Pd, Rh, etc. (1) has density 0.3–1.2 g/cm^3 and a surface area of 400–1200 m^2/g. The fuel cell also includes current collector plates, gas manifolds and a separator.

Battery for Electrical Energy Production

I. SEKINE *Japanese Appl.* 7/176,311

A battery for the continuous production of electrical energy in the home contains a fuel room within an auxiliary tank containing electrolyte which is pressure operated, and a Pd plated steel electrode, which acts as a catalyser. The battery has a longer life and a safety valve for pressure adjustment.

Solid State Polymer Electrolyte Fuel Cell

TOYOTA JIDOSHA K.K. *Japanese Appl.* 7/201,346

A fuel cell comprises a cathode catalytic reaction layer, which contains C particles and Pt, between an electrolyte film and a cathode. A fire resistant layer consisting only of C covers both sides of the catalytic layer. O_2 mixing with H_2 causes combustion to take place in the presence of the Pt and the heat generated on the peripheral edge of the cathode side of the catalytic layer is dissipated by the C particles of the fire resistant layer. Improved durability of electrolyte film is obtained.

Electrode Catalyst for Fuel Cell

FUJI ELECTRIC CO. LTD. *Japanese Appl.* 7/211,328

An electrode catalyst for fuel cells is manufactured from Pt particles supported on C which are heat treated at 1000°C in an inert gas. Pt is added to the dispersed C support from a Pt containing solution, is mixed, and a reducing agent is added to support the Pt on the C. The Pt is filtered, washed and dried to separate solid and finally heat treated. The fuel cell has enhanced generating power and a longer life. Deposited impurities are totally decomposed and scattered.

Apparatus for Production of Chlorine

MITSUI TOATSU CHEM. INC. *Japanese Appl.* 7/216,570

Production of Cl_2 from HCl acid in fuel cells comprises a Pt anode, a Pd cathode and an ion conductive membrane between two acid impregnated wool discs. Cl_2 is produced under mild conditions without yielding any by-products. The reaction energy can be brought in from outside when required.

CORROSION PROTECTION

Anticorrosion Coating Production

GENERAL ELECTRIC CO. *European Appl.* 671,486A

Modifying the initiation or propagation of cracks in the surface of metal components in H_2O-cooled nuclear reactors and associated equipment, comprises injecting a solution or suspension of thermally decomposable Pd compounds into the reactor H_2O. The Pd decreases the electrochemical potential of the metal component surface, and Pd ions and/or atoms are deposited or incorporated. The process reduces the electrochemical potential of stainless steel below the critical threshold. Pd doping is also effective against inter-granular stress corrosion cracking.

Hot Corrosion Protection

SERMATECH. INT. INC. *World Appl.* 95/23,243A

Hot corrosion protection for a Ni-base alloy is enhanced by enriching the surface by electroplating with Pt, heating to diffuse the Pt, and then diffusing molten Al and Si into the Pt-enriched substrate. This gives a Pt-enriched Si-modified corrosion resistant aluminide coating on the Ni-base alloy substrate. Also claimed is a Pt-enriched Si-modified aluminide coating on a refractory-containing Ni-base superalloy substrate. This coating is used for parts in gas turbine engines where temperatures are ≥ 12,000°C.

CHEMICAL TECHNOLOGY

Hydrogen Cyanide Production

E.I. DU PONT DE NEMOURS & CO. *World Appl.* 95/21,126A

Hydrogen cyanide is produced using an induction heated Pt group metal catalyst at 0.5–30 MHz frequency to the reaction temperature and then contacting with a mixture of NH_3 vapour and hydrocarbon gas to produce HCN, which is recoverable. The process is on a small scale, efficient and has low installation costs.

Production of *cis*-Substituted Cyclohexyl-Amine Derivatives

HOECHST-SCHERING AGREVO G.m.b.H. *German Appl.* 4,405,728

cis-Substituted cyclohexylamine derivatives (1) are prepared by reacting a cyclohexanone derivative with NH_3 and H_2 in the presence of a noble metal boride catalyst. The substituents may optionally be aliphatic, alicyclic, aromatic or heterocyclic groups. Derivatives (1) are useful as intermediates, for example for 4-amino- and 4-alkoxy-pyrimidines with insecticidal, acaricidal and fungicidal activity. The process gives high yields (> 90%) with high *cis* selectivity (~ 89–90%) at relatively low pressures and is suitable for either continuous or semi-continuous operation.

ELECTRICAL AND ELECTRONIC ENGINEERING

Multilayer Magneto-Optic Recording Medium

EASTMAN KODAK CO. *U.S. Patent* 5,436,072

A magneto-optic recording medium has a seed layer of amorphous oxide $Zn_{1-x}Sn_xO_{1+x}$, where x = 0.15–0.75, and a recording multilayer of alternating layers of Co and Pt on the seed layer. The seed layer is selected to improve the coercivity and squareness of the multilayer. The medium is used for magneto-optic disks, CDs and photo CDs and for cartridge use.

Photomagnetic Recording Medium

HITACHI LTD. *Japanese Appl.* 7/130,015

A photomagnetic recording medium includes Pt/Co multilayer film and/or Pt/CoNi multilayer films. At least one layer of Co or CoNi, of thickness greater than the transition metal element layer, in the multilayer film is formed, optionally in place of the multilayer film. The medium is suitable for high density recording using a short wavelength optical beam. It has improved magnetic field sensitivity.

Printed Wiring Board Manufacture

DAINICHI KOGYO K.K. *Japanese Appl.* 7/147,478

A method for the manufacture of printed wiring board uses a Ni plating layer on the conductor pattern deposited on a substrate which is laminated with Cu. Pd metal, in the form of an island, is deposited over the Ni plating layer. This method provides inexpensive soldering and reduces the wear effect.

Semiconductor Chip Terminal Formation

WORLD METAL K.K. *Japanese Appl.* 7/183,327

A semiconductor chip has input-output terminals formed from a base material, such as Al, which is first ground processed, using a Pd chloride solution. A thin Ni layer followed by a Pt group metal layer are then deposited onto the terminals by chemical plating. The terminals have enhanced solderability with improved bonding. The mounting contains no solder bump.

Perpendicular Magnetic Recording Medium

SONY CORP. *Japanese Appl.* 7/192,224

A perpendicular magnetic recording medium comprises laminations of a non-magnetic substrate, an undercoat layer and an alloy film based on Co/Cr. The undercoat layer is Pt. The medium is suitable for high density recording and has improved write/read characteristics. The Co/Cr magnetic film has 3094 cps peak intensity, since orientation and crystallisation are improved by the Pt undercoat layer.

Structure of Opto-Magnetic Recording Medium

KINSEISHA K.K. *Japanese Appl.* 7/192,328

An opto-magnetic recording medium has a substrate on which multiple recording film of varying thickness is accumulated. The substrate has a first dielectric film under a Pt layer. A first recording layer is arranged between the Pt layer and a Cr film. A second dielectric film with a laser beam reflecting coating layer is provided on a second recording film which is irradiated by a light beam of different wavelengths to write or read out from the disk. The medium reproduces information selectively and can simultaneously produce two amounts of recorded information.

Conductive Resin Paste

SUMITOMO METAL MINING CO. *Japanese Appl.* 7/192,527

Conductive resin paste contains globular powdered Ag with an average grain diameter of 0.01–5 μm; powdered Ru oxide of diameter 100–5000 Å; epoxy resin; a hardening agent; and a solvent consisting of an ester of a 6C or less monobasic acid and 6C or less primary or secondary alcohol, and glycidyl ether. The weight of the powdered Ru is 30–75 pts. wt./100 pts. wt. of the resin-based material. The paste is used for connecting semiconductor devices; it prevents malfunction of a photo-transforming switch, including a photocoupler, and gives stable conductivity.

Ceramic Wiring Board

MATSUSHITA ELECTRIC WORKS LTD. *Japanese Appl.* 7/212,009

Ceramic wiring board, for mounting electronic components, has a multilayer structure comprising a Cu layer and a Pd layer deposited by electroless plating. The thickness of the Pd layer is ≥ 1μm and is formed by electrolytic plating using current density of 1–20 A. The wiring board allows high resolution conduction patterns to be deposited and improves bonding properties. Heating up to 450°C prevents swelling.

Magnetic Recording Medium

TOSHIBA K.K. *Japanese Appl.* 7/235,034

A magnetic recording medium is formed by sandwiching vertically magnetised film containing Co-Pt alloy between Co oxide, nitride or carbide layers. The alloy is in crystalline form. The medium has increased saturation magnetisation, vertical magnetic anisotropy and coercive force. Electrical resistance is raised.

Conductive Paste for Through Hole Filling

NITSUKO K.K. *Japanese Appl.* 7/235,215

Conductive paste is formed by adding 0.1–3 wt.% Rh powder to electrically conductive powder. Initially, the through hole is filled with ingredients, such as Ag powder and then with Cu powder; the electrically conductive powder containing Rh powder is filled over this. The paste is highly shape stable and reliable, does not heat shrink, and has electrical resistance typical of small electric conductors. It does not crack and gives improved wiring density.

MEDICAL USES

Precious Metal Dental Alloy

J. M. NEY CO. *U.S. Patent* 5,431,875

An alloy for dental restorations comprises (wt.%): 60–95 precious metal(s) selected from: 60–85 Pd, 0–10 Pt, 0–5 Au, 0–12 Ag; 1–15 Sn; 2–7 Zn; 0.005–0.2 B; 0–2 Ga; 0–2 Co; 0–15 In; 0–0.2 deoxidant selected from Si, Ge, Mg, Al, Li and Ta; 0–1.0 grain refiners selected from Ru, Ir and Re. The alloy has a liquidus temperature of ≤ 1400°C, a tensile yield strength of ≥ 250 MPa, and an elongation of ≥ 2%. A claimed dental restoration method comprises casting the above metal alloy and firing a translucent porcelain coating upon it. The oxide film of the cast alloy does not discolour the translucent porcelain.

Diamino Cyclohexane Platinum Complexes

UNIV. TEXAS SYSTEM *U.S. Patent* 5,434,256

cis-1,4-Diaminocyclohexane Pt(IV) complexes and 1,2-diamino cyclohexane Pt(IV) complexes and their stereoisomers, containing Cl, propionate or butyrate; acetate, trifluoroacetate, propionate, butyrate, pentanoate, hexanoate or heptanoate, have been prepared. The compounds are antitumour agents active against leukaemia and solid tumours resistant to *cis*-platin and tetraplatin. The compounds are non-nephrotoxic and have a high activity.

Super-Elastic Spring

TOKIN CORP. *Japanese Appl.* 7/207,390

A super elastic spring for catheter guide wires comprises a Ti-Pd shape memory alloy with a martensitic transformation starting point of at least the service temperatures and ≥ 60% of the deformed strain spontaneously being elastically recovered at the service temperatures. It is additionally used for wires for straightening irregular teeth.

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