NEW PATENTS

METALS AND ALLOYS

Corrosion Resistant Titanium Alloy

NIPPON MINING K.K. *Japanese Appl.* 1/165,738

A corrosion resistant Ti alloy consists of more than 7 wt.% but less than 45 wt.% Ni, 0.01–5wt.% in total of at least one of Pt, Pd, Rh, Ir, Os, Ru and Au, and balance Ti and impurities. The alloy has improved corrosion resistance in a severe corrosive environment such as a high concentration aqueous solution of HCl or H_2SO_4, and is used for chemical facilities, heat exchangers, reaction vessels or electrodes.

ELECTROCHEMISTRY

Insoluble Anode for Electroplating

NIPPON STEEL CORP. *Japanese Appl.* 1/150,000

An insoluble anode for electroplating has a substrate, for example Ti, with a surface layer of spray-coated electroconductive ceramic(s) and a further layer of a Pt group metal or oxide. In an example the ceramic layer was 500 μm thick and the Pt-oxide film 30 μm thick. The electrode has reduced peeling off of its surface, longer life, and no flaws are generated on the plated surface of the object.

Electrode for Corrosion Prevention

SANYO ELECTRIC K.K. *Japanese Appl.* 1/162,785

An insoluble electrode consists of a Ti or Ti alloy substrate, plated with Pt or Pt-Ru, with an insulating coating material such as Teflon or polybutene having holes on the entire surface or woven in mesh form. The electrode surface is formed with hot water resistant, soft and flexible material, and the electrode is used for electric protection of hot water boilers.

Insoluble Electrode with Adhered Platinum Plate

TANAKA KIKINZOKU KOGYO *Japanese Appl.* 1/188,690

An insoluble electrode consists of a plate material of Ta, Ti or Nb, plated with 0.1–2 μm thick Pt, and adhered to a Pt plate by applying pressure. An electrode having a Pt plate tightly and uniformly adhered to the base plated can be manufactured easily without any special apparatus using this method.

Catalytic Electrode Used with Ion-Exchange Membrane

SASAKURA KIKAI SEIS. *Japanese Appl.* 1/208,489

An electrode consists of a porous base, an electroless plated layer of at least one of Pd, Rh, Ir and Ru on the surface, and an optional electrodeposited layer of PbO_2 or MnO_2 having an electrode catalyst function. The electrode is used for electrolysis by contacting with an ion-exchange membrane, for example zero-gap electrolysis used for producing H_2, O_2 and ozone from water, or halogens from aqueous halides.

Joint Structure for Solid Polymer Electrolyte Electrolysis

MITSUBISHI HEAVY IND. K.K. *Japanese Appl.* 1/208,490

A joint structure is made by forming a thin layer of metal on one surface of an ion-exchange membrane, depositing a metal layer on the other surface, and then a final metal layer on the first thin layer. Preferred surface joining metals are Pt, Pd, Rh, Ir and Ru, which have corrosion resistance and are able to produce H_2, O_2 or Cl_2. The joint structure is free from cracks and peeling off.

Electrochemical Apparatus for Oxygen-Hydrogen Generators

JAPAN STORAGE BATTERY *Japanese Appl.* 1/222,082

Electrochemical apparatus has an ion-exchange membrane catalyst electrode body which consists of an ion-exchange membrane with platinum group metals attached to both sides. Water is supplied to the anode or both electrodes while applying DC to effect electrolysis, having a diode and switch included between the anode and the DC power source.

ELECTRODEPOSITION AND SURFACE COATINGS

Palladium Catalyst Solution for Electroless Plating

HITACHI CHEMICAL K.K. *European Appl.* 328,263A

An aqueous catalyst solution for electroless plating consists of 1 mol of a divalent Pd compound, 1–20 mol of a lower alkylamine, and 0.5–10 mol of an aminopyridine, also with at least 0.001 mol of a silane coupling agent. The solution is of pH 7–14, and gives uniform deposition and high throwing power of Cu on the inner walls of through-holes in printing wiring boards, without causing hollowing phenomena.

Tin-Palladium Sensitising Activator

SOLUTION TECHN. SYST. *World Appl.* 89/6,710A

A sensitising activator for chemical plating consists of a colloidal dispersion of $SnCl_2$ and $PdCl_2$ in aqueous NaCl solution containing HCl or a polyvalent acid in dilute concentration, and may also contain an aromatic aldehyde. The activator is used especially in chemical plating through-holes in a multilayer printed circuit board, and provides good permeation and adhesion to a substrate.

Electroplating Bath for Palladium-Nickel Alloy Deposition

DU PONT DE NEMOURS CO. *U.S. Patent* 4,846,941

A bath for electrodeposition of a Pd-Ni alloy contains salts of Pd and Ni, preferably Pd(II) ammine chloride and Ni ammine sulphate, sulphate or chloride; ammonium sulphate and at least 15 ppm of iodate to stabilise the deposit composition.

Production of Palladium Coated Silver Powders

SUMITOMO METAL MINI K.K. *Japanese Appl.* 1/198,403

The surface of spheroidal Ag powders of particle size 0.1–1.2 μm is given a Pd coating of more than 5 wt.% by suspending the Ag powder to make a slurry, adding a reducing agent, and adding 10–100 g/l Pd as Pd dichloro diamide solution to deposit Pd. The Pd coated Ag powders exhibit high heat resistance, good conductivity, high Ag separation temperature, and may be added to an organic vehicle to produce a conductive coating material.

Lightweight Electrode for Electroplating

DAISO CO. LTD. *Japanese Appl.* 1/208,499

An electrode consists of a porous metal anode coated with a layer of a Pt group metal oxide, preferably at least one of Pd, Rh, Ir, Os and Ru. The electrode is used for electroplating in a continuous plating bath and is lightweight and convenient for handling. Gas generation in the bath is more easily dissipated, thus improving plating quality and cost reduction.

Manufacture of Metal Plated Ceramic Moulding

TODA KOGYO *Japanese Appl.* 1/222,063

A metal plated ceramic moulding is made by dipping into a Pd-hydrosol containing a water soluble polymer or with surfactant(s), to absorb Pd colloids into the surface of the ceramic moulding, followed by rinsing and chemical plating. The method is used for sensors, printed circuit boards, high-frequency resonators, and capacitors.

APPARATUS AND TECHNIQUE

Temperature Controlled Thermal Radiation Source

SIEGER LTD. *British Appl.* 2,211,379A

A temperature controlled thermal radiation source includes a wire filament source-coil integral with a sensor wire coil for sensing and controlling the temperature of the source. The sensor wire is of ZGS-Pt, ZGS-Pt-5% Rh, ZGS-Pt-10% Rh, or any similar stable material. The source has high sensitivity to the effect of the environment on temperature, enabling excellent temperature stability.

Plate-Shaped Oxygen Sensor

JAPAN ELEC. CONTROL SYST. *European Appl.* 331,050A

An O_2 sensor for an I.C. engine consists of an air cavity between a substrate and an O ion conducting solid electrolyte layer, electrodes on the electrolyte surfaces contacting air and exhaust gas, respectively, and a NO_x reducing catalyst preferably of Rh, Ir or Ru supported on TiO_2 on the surface contacting the exhaust gas and its electrode. A plate-shaped O_2 sensor is provided with a uniform catalyst reaction surface.

Low Power Consumption Catalytic Gas Detector

SIEGER LTD. *European Appl.* 334,614A

A catalytic gas detector having low power consumption consists of a catalytically coated resistive heating sensing element formed as a conductive track on a substrate, that is, Pt on a resistor in a Wheatstone bridge on nitrided Si. The gas detector is cheap to manufacture, has lower power consumption than usual, and many detectors may be formed on the same wafer.

Solid-State Thin Film Moisture Sensor

ROSEMOUNT INC. *World Appl.* 89/7,264A

A thin film moisture sensing element is prepared by forming a SiO_2 layer on a Si substrate, covering part with developed photoresist, depositing Ti, Ni or Cr on at least part of the uncovered oxide and a layer of Pt, Rh, Ir or Au on the first metal layer, removing the remaining photoresist, applying aqueous H_3PO_4 to the oxide layer, and drying.

Combustible Gas Detector with Platinum Sensor

MINE SAFETY APPLI. CO. *U.S. Patent* 4,854,155

A catalytic gas detector circuit is a bridge circuit with four branches, one of which is a catalytic gas sensing element reactive to combustible gases. The sensor is a coiled Pt filament embedded in a catalytic bead. A current changer is also present which temporarily increases the current, to exceed the operating temperature of the gas sensing element.

Iridium-Zirconium Crucible Material

TANAKA KIKINZOKU KOGYO *Japanese Appl.* 1/119,595

A crucible material useful for high temperature single crystal growths consists of pure Ir with an admixture of 200–10,000 ppm Zr. Preparation of the material involves melting, casting at 1500°C, and hot rolling at 1000°C. The material is hard, and the constituent fine crystal grains are free from coarsening at high temperature and also from crack development at the grain boundaries.

Crucible for Preparing Single Crystals

MURATA MFG. CO. *Japanese Appl.* 1/131,095

A crucible consisting of Pt-Au alloy with 1–30 at.% Au is used for producing oxides with low melting point (not higher than 1400°C) by the Czochralski method. Using the crucible, inclusion of bubbles and cracks is avoided, and single crystals have diameter not less than 60% of the crucibles inner diameter.

Iridium Crucible Allowing Uniform Heating

TANAKA KIKINZOKU KOGYO *Japanese Appl.* 1/145,392

An Ir crucible with bottomed cylindrical shape and a height diameter ratio of up to 2 is produced from an Ir disc 0.1–3.5 mm thick and 5 times the crucible diameter by pressing against a columnar metal mould while rotating and heating at 800°C or above.

Double Crucible for Producing Noble Metal Granular Lumps

TANAKA KIKINZOKU KOGYO *Japanese Appl.* 1/150,788

A crucible for producing Pt, Pt alloy, Pd or Pd alloy metal granular lumps of 8 mm or less diameter consists of an inner ZrO_2 crucible with a nozzle and an outer crucible of alumina and silicon oxide. The double crucible prevents the crucible and nozzle from damage by thermal impact.

Semiconductor Pressure Sensor

FUJIKURA CABLE WORKS K.K. *Japanese Appl.* 1/173,847

A metal thin membrane layer consisting of at least a Ti layer up to 2500 Å thick and a Pt layer 1000–2500 Å thick, is formed on the bottom surface of a mount. A sensor chip is bonded on the upper surface of the mount which is then adhered onto a base. The pressure sensor has high reliability, and by controlling the Pt and Ti layer thicknesses, leak failure of the sensor can be prevented.

Tin Oxide Semiconductor Gas Sensor Device

RIKEN CORP. *Japanese Appl.* 1/189,553

A gas sensitive membrane has 0.1–20 wt.% of at least one of Pt, Pd and Ir finely dispersed in Sn oxide by simultaneous deposition of the metal and Sn oxide by a sputtering or heat deposition process. The two-phase structure thin membrane produced is sensitive to various reduction gases at below 200°C, and a small, high sensitivity gas sensor device can be easily produced at low cost.

Bubble Removal Apparatus

MITSUBISHI HEAVY IND. K.K. *Japanese Appl.* 1/189,555

Apparatus for removing bubbles from an electrophoresis solution consists of an electrophoresis bath, a circulation channel, and a reaction bath having a catalyst layer with supported Pd. This separation system can be used for electrophoresis for separation and purification of materials in space.

Oxygen Sensor with Platinum Electrodes

TOYOTA JIDOSHA K.K. *Japanese Appl.* 1/203,964

An O_2 sensor used for example for automobiles, has a sensor body formed from solid electrolyte ceramic powder, with a first electrode layer formed from paste material of the same powder and Pt powder, and a second electrode layer formed from paste material of 0–30 wt.% of the ceramic powder and 70–100 wt.% of Pt powder. The electrodes have good low temperature activity and response speed.

Carbon Monoxide Sensor for Alarm Equipment

SEIKO K.K. *Japanese Appl.* 1/219,660

A catalytic combustion type CO sensor has a circuit with an active part and a compensation part made of a coil of Pt or Pt type alloy wire, respectively. By controlling the V-R (or T) characteristics of these parts the zero drift can be prevented or decreased, giving a CO sensor for use in various environments.

Assessment of Polyurethane Ageing

AKAD. WISSENSCHAFT DDR *East German Patent* 267,798

Visual assessment of the ageing of naturally or artificially weathered polyurethanes involves observing the intensity of colour produced by treating the material with Os tetroxide or uranyl acetate, for 4 hours to 14 days, at 20–70°C. The invention provides a simple, direct process for assessing the ageing of polyurethane sheet or film, as required in manufacturing and applications such as paint production.

JOINING

High Temperature Braze Containing Palladium

GENERAL ELECTRIC CO. *European Appl.* 329,954A

A brazing filler metal with liquidus temperature over 700°C preferably contains 20–80 wt.% Pd, 2–13 wt.% Cr, 1–4 wt.% B and balance Ni. The brazing composition is used to bond a self-bonded porous diamond compact to a cemented carbide support or similar compact, in the manufacture of grinding, abrading and cutting tools. The Cr in the braze gives bond strengths over 90 kpsi for a surface which has been difficult to bond to.

Brazing Alloy Containing Palladium

GTE PRODUCTS CORP. *U.S. Patent* 4,839,141

A brazing alloy consists of 15–35 wt.% Pd, 5–30 wt.% Au, 10–30 wt.% Ni, 20–48 wt.% Cu, and 5–25 wt.% Mn, and has a brazing temperature of 1025–1080°C, a solidus temperature >1000°C, and a liquidus temperature >1018°C. The alloy has good gap filling and high temperature properties, and is used in joining superalloy metals such as Inconel 718; the alloy brazing at the solution heat treating temperature of the superalloy.

Brazing Alloy for Bonding Superalloy Honeycomb Structure

GTE PRODUCTS CORP. *U.S. Patent* 4,853,291

A brazing alloy consists of 15–35 wt.% Pd, 5–30 wt.% Au, 10–30 wt.% Ni, 20–48 wt.% Cu, and 5–25 wt.% Mn, and has a solidus and liquidus above 1000 and 1018°C, respectively. The alloy brazes at the superalloy solution heat treating temperature, has good gap filling and high temperature properties, and is used for a panel with a honeycomb stucture of thin superalloy metal sandwiched between and brazed to 2 sheets of superalloy metal.

Brazing Filler Material

HITACHI K.K. *Japanese Appl.* 1/118,394

A brazing filler material uses Al-Pt or Al-Pd alloy containing 0.1–5 wt.% Pt or Pd as an insert alloy for jointing, and is best suited for joining Ti and Ti alloy by brazing and liquid phase diffusion joining. Deterioration of joining material can be prevented and influences of gas can be eliminated, improving the corrosion resistance of the weld zone.

Palladium-Silicon-Nickel-Chromium Solder Material

HITACHI K.K. *Japanese Appl.* 1/178,399

A solder material for joining graphite materials consists of 10–30 wt.% Pd, 1–10 wt.% Si, and balance Ni and Cr. A strong joint of graphite materials is obtained, for severe conditions such as high temperature. In an example the solder material was used to join a graphite plate and a Mo plate, giving an average shear strength of 5 kg/mm^2.

HETEROGENEOUS CATALYSIS

Ruthenium Hydrogenation Catalyst

HULS A.G. *European Appl.* 324,190A

A catalyst consisting of 0.1–5 wt.% Ru on a support of BET surface area 70–280 m^2/g and average pore diameter 10–320 Å is used with alkali and alkaline earth metal promoters to give high yields of 4,4′-diaminodicyclohexyl methane by hydrogenation of for example 4,4′-diaminodiphenyl methane. The hydrogenation reaction is at 100–190°C and 50–350 bar in at least one fixed bed reactor, giving a product with a trans-trans isomer content of 15–40 wt.%.

Production of Diphenylamines

BAYER A.G. *European Appl.* 325,132A

Supported catalysts having a total noble metal content of 0.05–5 wt.% made up of 10–90 wt.% Rh, and Pt, Pd or Ir; 1–6 wt.% alkali hydroxide and 1–6 wt.% alkali metal sulphate are used in the production of diphenylamines by reaction of dicyclohexylamines at 250–450°C and 1–20 bar. Using this catalyst diphenylamines can be produced in high yield by a simple process, with easily processed products.

Enhanced Activity Dehydrogenation Catalyst

LABOFINA S.A. *European Appl.* 328,507A

A catalyst consists of an Al_2O_3 support with 0.1–2 wt.% of at least one platinum group metal, 0.1–2 wt.% of metal(s) of the tin group as co-catalyst and 0.5–5 wt.% alkali or alkaline earth metal as promoter. Preparation involves 2 treatments of the support with a platinum group metal compound, and several calcination steps, giving a catalyst of enhanced activity for dehydrogenation, dehydrocyclisation and hydrogenation reactions.

Palladium-Rhodium Exhaust Purification Catalyst

ENGELHARD CORP. *European Appl.* 329,302A

A catalyst for purification of I.C.E. exhaust gas consists of Pd, Rh, active Al_2O_3, plus compounds of Ce, Sr and Zr deposited on a monolithic support. The Ce, Sr and Zr compounds are for example Ce oxide, and oxides and carbonates of Sr and Zr, and inhibit sintering of Pd and Rh, and promote adsorption of gases. The catalyst is effective at low temperatures even after exposure to high temperatures.

Preparation of Supported Metal Catalysts with Large Surface Areas

TANAKA KIKINZOKU KOGYO *European Appl.* 329,628A

Catalysts are prepared by reducing Pt and/or Pd salt solutions to the metal by addition of a weak reducing agent containing S, for example $Na_2S_2O_3$, $K_2S_2O_3$ or $(NH_4)_2S_2O_5$, and adsorbing the metal onto a C, SiO_2 or Al_2O_3 support. The products have large surface areas leading to increased activity, and are durable against sintering. The deposited metal particle size is smaller and more uniform compared to use of strong reducing agents.

Platinum and/or Palladium Oxidation Catalyst

PHILLIPS PETROLEUM CO. *European Appl.* 330,224A

A catalyst is prepared by contacting a support with a Pt and/or Pd compound solution of at least pH 5 and heating and drying to convert to the oxide or metal. The catalyst may contain an Fe compound. Uses include oxidation of CO to CO_2 in the presence of free O_2, for example in I.C.E. exhaust gases, in air, or to combine CO and O_2 in CO_2 lasers, and also manufacture of isotopically labelled CO_2.

Single Step Process for Production of Mono-Olefins

BRITISH PETROLEUM PLC *European Appl.* 332,289A

Production of mono-olefins by oxidative dehydrogenation of paraffinic hydrocarbons involves partial combustion of a mixture of hydrocarbons and O_2-containing gas in contact with a catalyst of platinum group metals supported on a monolith. A mixture of ethane, propane or butanes can be converted into mono-olefins in a single step process, especially from a gaseous mixture of mainly ethane resulting from the separation of CH_4 from natural gas.

Palladium Dehydrogenation Catalyst for Phenol Preparation

MITSUI PETROCHEM. IND. K.K. *European Appl.* 332,380A

Phenol and/or a cyclohexanone are prepared by dehydrogenation of cyclohexanone and/or cyclohexanol, respectively, in the liquid phase in the presence of a solid catalyst consisting of Pd on a carrier of activated C and/or a hydrotalcite. Phenol is useful as an intermediate in the production of phenolic resins, and cyclohexanone is useful as an intermediate for high molecular weight compounds such as nylon and polyesters.

Catalysts for Hydrocarbon Steam Reforming

A. IGARASHI *European Appl.* 333,037A

Catalysts for steam reforming of hydrocarbons consist of (a) 0.01–5 wt.% Ru on ZrO_2, (b) 0.1–5 wt.% Rh and/or Ru on partially stabilised ZrO_2, and (c) 0.1–5 wt.% Rh and/or Ru, and 0.1–10 wt.% Ni, La, Pr or Nd on ZrO_2. The catalysts have high activity, heat resistance and mechanical strength.

Novel Exhaust Treatment Catalyst with Sulphur Tolerance

SOC. FR. PROD. CATALYSE *European Appl.* 333,559A

A novel catalyst for treatment of I.C.E. exhaust gas consists of a substrate, and a porous layer of Al_2O_3, rare earth oxide(s) and a spinel-type compound with a specific surface of at least 50 m^2/g, impregnated with one or more precious metals from Pt, Pd, Rh and Ir. The catalyst and the support have good thermal stability, and tolerate the presence of S compounds in the exhaust gas.

Highly Selective Rhodium Hydrogenation Catalyst

AIR PRODUCTS & CHEM. INC. *European Appl.* 335,272A

Catalytic hydrogenation of aromatic amines to their ring hydrogenated counterparts is improved by using a Rh catalyst supported on TiO_2. Impure or crude aromatic amines can be used without poisoning the catalyst, relatively low pressures are required which reduces costs, the processs is highly selective, the products are free from by-product oligomers, and the catalyst can be used over a period of time with little maintenance.

Three-Way Catalyst for Emissions Control

SVENSK EMISSIONSTEK *European Appl.* 335,847A

A catalyst for oxidation of hydrocarbons and CO and/or reduction of NO_x consists of a carrier with at least 2 washcoat layers containing Al_2O_3, one or more metal oxides with a promoting effect, and Pt, Pd and/or Rh. The layers have different noble metal contents, with for example 75–100% of the total Pt, and also Rh in one layer. The catalyst is used for treatment of combustion engine exhaust gases.

New Partially Reduced Ruthenium Oxide Catalysts for Methanation

GAS RESEARCH INST. *U.S. Patent* 4,847,231

The catalysts consist of 1–15 wt.% of a mixture of 10–90 mol% Ru and 10–90 mol% RuO_x where $0<x<2$, dispersed on a support preferably of TiO_2. The catalysts based on semiconducting oxides are useful for photochemical conversion of CO_2/H_2 mixtures to CH_4, are active at low temperatures and pressures such as 25–90°C and 1 atm, and give >99% selectivity. Catalysts based on Al_2O_3 or zeolite are useful for dark methanation.

Catalyst for Reducing Diesel Soot Ignition Temperature

ALLIED-SIGNAL INC. *U.S. Patent* 4,849,399

A catalyst composite consists of at least one catalytic metal deposited on at least one S-resistant inorganic oxide, which is deposited on a diesel soot filter. The catalytic metal can be Pt or Pd at 5–250 g/ft^3 or Rh at 2–70 g/ft^3 filter volume. The catalyst ignites the soot in diesel engine exhaust at a lower temperature than prior catalysts, and by its use the exhaust temperature of the engine may be reduced.

Electrically Heated Catalytic Converter for Air Pollution Control

M. JOHN *U.S. Patent* 4,852,530

A three-way catalytic converter in the exhaust consists of a base portion and an electrically heated rod and fins, covered with a thin coating of Pt, Pd and Rh. Low temperature air pollutants flowing through the converter contact the heating rod and heating fins, which causes a rapid increase in exhaust temperature, so that oxidation of hydrocarbons and CO, and reduction of NO_x take place on the catalytic surfaces.

Cyclic Gas Desulphurisation Process

INST. FRANCAIS DU PETROLE *U.S. Patent* 4,855,117

A sorbent consisting of 1–50 wt.% MgO on Al_2O_3 containing 0.001–0.1 wt.% Pt or Pd is contacted with SO_2-containing gas in the presence of O_2, at 350–750°C, to fix SO_2 as $MgSO_4$. To regenerate MgO the sorbent is contacted with a reducing gas of S vapour, at 500–750°C, giving a gas effluent containing SO_2, but free from SO_3 and H_2S. The process is used to treat fumes from thermal power plants, boilers and Claus units.

Improved Heat Resistance Palladium Combustion Catalyst

HITACHI K.K. *Japanese Appl.* 1/130,733

A combustion catalyst is produced by mixing 0.1–5 wt.% of Pd fine powder having 1000–5000 Å particle size with a porous support such as γ-Al_2O_3. Volatile Pd fine powders smaller than 1000 Å are not included, and a catalyst with improved heat resistance is obtained for use at 800–1500°C. In an example CH_4 was combusted with the catalyst at 450°C with 83% efficiency.

Catalysts for Automobile Exhaust Purification

TOYOTA JIDOSHA K.K. *Japanese Appls.* 1/139,144–45

Catalysts for cleaning automobile exhaust consist of (a) a reducing catalyst on the inflow side obtained by ion-exchanging zeolite with Pt, Pd, Rh, Ir, Ru or others, and an oxidising or ternary catalyst on the outflow side having Pt, Pd, Rh, Ir, Ru or others supported on Al_2O_3; or (b) a catalyst obtained by blending a reducing catalyst and an oxidising catalyst as in (a). The catalysts have good performance for removing CO, hydrocarbons and NO in an atmosphere containing excess O_2.

Oxidation of Polyoxyethylene Alkyl Ethers

KAWAKEN FINE CHEM. K. *Japanese Appl.* 1/149,752

Polyoxyethylene alkyl ethers are oxidised by reaction of a 10–50 wt.% non-alkaline aqueous solution with O_2 containing gas, at 30–90°C, by passing through a reactor packed with granular Pt-Pd/C catalyst. High quality polyoxyethylene alkyl ether acetic acid is prepared efficiently in a short time by this procedure; useful as a surfactant, modifier of functional polymers, or a material of biopolymers.

Production of Saturated Higher Ketones

MITSUBISHI KASEI CORP. *Japanese Appl.* 1/151,533

Higher ketones are produced by the one-step reaction at 30–150°C of lower ketones and H_2 in the liquid phase in the presence of a 0.001–10 wt.% Pd/ZrO_2 catalyst, in which the ZrO_2 has substantially neutral surface acid-base strength. Saturated higher ketones can be thus obtained economically.

Moisture Resistant Catalyst for Carbon Monoxide Removal

NIIKI UNIVERSAL K.K. *Japanese Appl.* 1/159,058

A catalyst for removing CO consists of Pt or Pd and at least one of Fe, Co, Ni, Mn, Cu, Cr, Sn, Pb or Ce on an Al_2O_3 support having no pores under 110 Å in diameter. The catalyst has good moisture resistance, so activity is not lowered by moisture in the air during use.

Waste Gas Purification Catalyst

TOYOTA CENT. RES. & DEV.

Japanese Appl. 1/168,343

A catalyst used to remove CO, hydrocarbons or NO_x from car exhaust is a mixture of a perovskite double oxide, a heat-resisting double oxide which may contain Ti, Zr or Hf with other metals, and precious metals selected from Pt, Pd, Rh, Ir, Os and Ru. The catalyst can be used above 900°C for a long period without reaction between the support and the double oxide or sintering the perovskite double oxide.

Exhaust Gas Purification Catalyst

TOYOTA CENT. RES. & DEV.

Japanese Appl. 1/171,625

A catalyst system used to remove NO_x, CO and hydrocarbons from internal combustion engine exhaust gas has a first bed of Cu/zeolite catalyst, and a second bed of a ternary catalyst containing Pt/Pd, Pt/Rh, or Pt/Pd/Rh, and optionally Ce. The first bed is used for low temperature exhaust gas (lean burn), and the second when it is 600–700°C (rich burn).

Diesel Exhaust Purification Catalyst

NIPPON SHOKUBAI KAGAKU *Japanese Appl.* 1/171,626

A catalyst for purification of Diesel engine exhaust gas consists of a heat resistant three dimensional structure such as ceramic foam or ceramic honeycomb having alternately sealed ends on both sides, carrying porous inorganic material and 0.1–7.0 g/l of one or more of Pt, Pd and Rh.

Palladium-Rhodium Exhaust Gas Purification Catalyst

CATALER KOGYO K.K. *Japanese Appl.* 1/184,037

A catalyst for purifying exhaust gas is produced by supporting Pd on a refractory porous support, immersing in a Rh solution prepared by heating $(NH_4)_3(Rh(NO_2)_6)$ in an acid solution and ageing to support Rh, and firing at 200–500°C to decompose the Rh salt. The catalyst is used to decompose CO, hydrocarbons and NO.

Production of Hydrogenated Petroleum Resins

ARAKAWA KAGAKU KOGYO *Japanese Appl.* 1/190,704

Hydrogenated petroleum resins are produced from gaseous H_2 and molten petroleum resins containing aromatic nuclei by continuous hydrogenation of the aromatic nuclei over 0.2–10 wt.% Pd on Al_2O_3 catalysts. A cheap Pd catalyst is used, the reaction is easily controlled, and resins are produced easily having a hydrogenation rate of 50–70%.

Iridium Catalyst Used in Recovery of Anilines

MITSUI PETROCHEM. IND. K.K.

Japanese Appl. 1/203,353

A catalyst of 0.1–10 wt.% Ir on active charcoal is used in the recovery of anilines by reaction of a 5–50 wt.% solution of condensation products of aniline and formaldehyde with H_2, in an ether group solvent. Reaction is at 200–350°C, under H_2 pressure of 10–100 kg/cm^2, for 0.2–5 hours. Aniline, toluidine and xylidine are recovered in high rates from the condensation by-products, and the aniline is reused.

Preparation of Hydrocarbons from Carbon Dioxide

AGENCY OF IND. SCI. TECH.

Japanese Appl. 1/221,332

Hydrocarbons are prepared by irradiating CO_2 in water with light of more than 310 nm wavelength, in the presence of a suspension of 1–5 wt.% Pt on TiO_2 in water. Preferably the TiO_2 is an anatase type TiO_2 having a large specific surface area. The preparation gives methane and ethane from CO_2 using water as a reducing agent, in contrast with H_2 in the prior art.

Catalysts for Vinyl Acetate Preparation

HOECHST A.G. *German Appl.* 3,803,900

A process for the preparation of vinyl acetate from ethene, acetic acid and O_2 in the gas phase uses supported catalysts of Pd or its compounds, Au or its compounds, or Cd compounds. The support is of aerated SiO_2 or Al_2O_3/SiO_2 of surface area 50–250 m^2/g formed into cylinders with rounded ends. Slightly higher selectivity and greatly increased productivity are found with the aerated catalyst and new shaping.

Palladium Catalysts for Butynediol Reduction

VEB. LEUNA-WERK ULBRICHT

East German Patents 265,394–96

Continuous processes for the hydrogenation of 1,4-butynediol to 1,4-butanediol are effected (a) in one stage using a Pd catalyst which can be a boehmite-supported Pd complex salt with 0.1–5 wt.% Pd, or (b) in two stages, the first using a boehmite supported Pd catalyst and the second using a supported Ni catalyst. Excellent yield and conversion are achieved, the processes are simple, safe and economical, and a high purity product is obtained.

HOMOGENEOUS CATALYSIS

Osmium Catalyst for Oxidation of Alkanes

RHONE-POULENC CHIM. *European Appl.* 331,590A

Alkanes are oxidised to a mixture of alcohols and ketones by an organic hydroperoxide at >20°C with a catalyst of Os or a compound of Os. For example cyclohexane gives cyclohexanol and cyclohexanone which are intermediates for adipic acid and caprolactam, and ethylbenzene gives 1-phenylethanol and acetophenone for production of styrene. The catalyst is more heat-stable, easier to obtain, and can be recycled.

Acetic Acid Production by Isomerising Methyl Formate

DAIREN KAGAKU KOGYO *Japanese Appl.* 1/153,652

Acetic acid is produced by heating methyl formate at 160–240°C and a pressure of 100–1500 psig in the presence of an organic solvent, CO, a soluble Rh catalyst, a halogen-containing accelerating agent and at least one accelerating agent of an Fe or V compound. The method produces acetic acid with a yield of at least 95%.

Preparation of High Purity Monochelate Palladium Complexes

UBE INDUSTRIES K.K. *Japanese Appl.* 1/157,992

Pd nitrate monochelate complexes of formula $LPd(NO_3)_2$, where L is a basic bidentate ligand, are prepared by reaction of Pd nitrate bischelate complexes of formula $L_2Pd(NO_5)_2$ with equivalent or more molar Pd nitrate. Highly pure and active monochelate complexes are readily prepared by this method, and are used as catalyst in the preparation of 3,3′, 4,4′-biphenyl tetracarboxylate by oxidative coupling of *o*-phthalate.

Phenol Preparation Using Palladium Catalyst

IDEMITSU PETROCHEM. K.K. *Japanese Appl.* 1/211,540

Phenol is prepared by oxidation of benzene with O_2 containing gas at 100–300°C, under 0–50 kg/cm²G, using a catalyst consisting of Pd, metal nitrate(s) and fatty acid Li salt(s), in molar ratio 1:0.5:0.5–1:5:10, respectively. Phenol is prepared under milder conditions than those of prior art, and in higher yield.

New Palladium Catalysts for Polymer Preparation

SHELL INT. RES. Mij. B.V. *Dutch Appl.* 88/349

New catalyst compositions contain a Pd compound, preferably a carboxylate such as Pd acetate, an anion of an acid with pKa less than 6, a diphosphine, and optionally a promoter such as a 1,4-quinone. The catalysts are used for preparation of polymers by polymerising a mixture of CO and an olefinically unsaturated compound, preferably ethylene or an ethylene/propylene mixture, and give polymers with higher molecular weight.

FUEL CELLS

Platinum Alloy Carbide Catalyst for Fuel Cell Electrode

TANAKA KIKINZOKU KOGYO *European Appl.* 329,626A

A catalyst used as a fuel cell electrode catalyst consists of carbides of Pt and one or more of Ni, Co, Cr and Fe on C supports. Preparation is by depositing Pt on the C supports by reducing a Pt containing ion in solution, adding one or more of Ni, Co, Cr and Fe, alloying, and carburising the metals. Superior catalyst performance is obtained because the metals are firmly fixed to the C supports by the carburising.

CORROSION PROTECTION

Palladium-Nickel Alloy Intermediate Layer

LPW-CHEMIE G.m.b.H. *German Appl.* 3,809,139

An aqueous electrolyte containing Pd and Ni amines with 2–20 g/l Pd and 5–30 g/l Ni plus conductive salt and organic additives, is used to form an intermediate layer between a low corrosion resistant metallic substrate and a plasma vapour deposited coating. The Pd:Ni ratio is adjusted to give a precipitated alloy with 30–90 wt.% Pd. The layer is used for corrosion protection, for metallised plastic components, and gives improved properties to the final coating.

CHEMICAL TECHNOLOGY

Noble Metal-Tin Oxide Catalyst for Carbon Dioxide Lasers

NAT. AERO & SPACE ADMIN. *U.S. Patent* 4,855,274

A ceramic support such as high surface area SiO_2 spheres is coated from solution with metastannic acid and Pt or Pd, followed by evaporation and drying to give Sn(IV) oxide. Catalyst preparation is easy and chloride free, very thin layers of tin oxide and noble metal are obtained, there is no pore blocking in the support and higher surface areas are achieved. The products are used in CO_2 lasers and are efficient at room temperature.

Refining Hydrogen Using a Palladium Membrane

NIPPON PIONIX K.K. *Japanese Appl.* 1/145,303

Refining H_2 involves introducing raw H_2 gas containing impurities into a H_2 purifying cell in which H_2 permeable Pd tubes are installed, and making H_2 flow out of the purifying cell. The method involves purging the impurities in the purifying cell.

Metal Containing Fibre Production

NICHIBI K.K. *Japanese Appl.* 1/148,873

A metal containing fibre is produced by spinning a solution of polyvinyl alcohol and a water soluble metal salt, preferably of Pt, Pd, Rh, Ir, Os, Ru, Au, Ag or others, and then reducing. A polyvinyl alcohol fibre is obtained having electric, magnetic, catalytic or bioactive functions depending on the metal in it.

Production of Flaky Noble Metal Granules

TANAKA KIKINZOKU KOGYO *Japanese Appl.* 1/152,205

Flaky granules of Pt, Pd or similar noble metals can be produced by continuously dropping moltem noble metal into water through the nozzle of a high frequency induction furnace, and crushing it into flaky granules in the water by a rotary conical rotor before solidification. The granules can be produced easily with high yield, and have a mean grain size of about 3.5 mm and a mean thickness of about 2 mm.

Reservoir Furnace for Producing Granular Noble Metal Lumps

TANAKA KIKINZOKU KOGYO *Japanese Appl.* 1/155,188

A reservoir furnace used for producing Pt or Pd noble metal granular lumps contains a ZrO_2 crucible covered with C and having a nozzle at the centre of the base. The crucible is in a high frequency induction heating unit, and is heated to high temperatures to maintain molten Pt or Pd metal.

Palladium-Silver Separation Membrane for Hydrogen

ISE KAGAKU KOGYO K.K. *Japanese Appl.* 1/164,419

A membrane is produced using a porous material having thermal resistance, forming a Pd membrane layer on the surface by chemical galvanising, forming a Ag membrane layer by chemical galvanising, and heat treating the layered material. The membrane layer has durability to degradation and cracking by use at low temperature (up to 300°C), and is used for separation of H_2, to give 100% purity.

Silver Halide Photographic Photosensitive Material

FUJI PHOTO FILM K.K. *Japanese Appl.* 1/167,746

The material has at least one emulsion layer composed of Ag halide particles with at least 90 mol % Ag chloride, and includes a small amount of a Rh salt; with <0.15μm particle size. A hydrazine derivative is also included in the emulsion layer and there is at least one non-photosensitive hardened upper layer. A photosensitive material for daylight utilising contrast by a hydrazine compound can be obtained.

ELECTRICAL AND ELECTRONIC ENGINEERING

Semiconductor Device Used for LSI

HITACHI K.K. *European Appl.* 326,018A

A semiconductor device with improved conductor films has a substrate with a plurality of Al-Pt or Al-Pd alloy films, and a protective film including the noble metal oxide on the conductor side wall. A claimed device has conductor line width of 1.3μm or less, up to 5 wt.% Pd, and Pd oxide in the protective film. The devices are used for large scale integration (LSI), and have improved fine pattern formation in Al films, and high resistance to electromigration.

High Resistance Data Storage Medium

PLASMON LTD. *European Appl.* 328,368A

A data storage medium has a surface region which includes a layer of heat-sensitive material with a textured surface pattern. The pattern is coated with a thin film of an alloy having at least one of Pt, Pd, Rh, Ir, Os or Ru with at least one other metal from specified groups. The device is used as an optical data storage disc, and uses a laser beam at relatively high power, while having high resistance and little or no deterioration.

Magnetic Thin Film for Recording Medium

SONY CORP. *European Appl.* 330,116A

A new magnetic film containing oxide(s) of Co, Pt, B and at least one of Ti, Zr, V, Cr, Nb, Mo, Ta and W provides high magnetic coercive force, high saturation magnetic flux density and a high perpendicular anisotropic magnetic field, even at a thickness <5000 Å. The film may be formed at a temperature as low as room temperature which allows a cheap substrate to be used when producing a magnetic recording medium.

Thin Film for High Density Magnetic Recording

HEWLETT PACKARD CO. *European Appl.* 334,488A

A magnetic film consists of an alloy of 10–20 at.% Pt, 10–20 at.% Cr, and balance Co, on a layer of Cr. The medium is for high density longitudinal recording, has reduced noise, good corrosion resistance, and independent adjustment of film coercivity (which is governed by the % Pt) and the saturation magnetisation-thickness product (governed by the % Cr and Pt and the film thickness).

Conductive Fibres Used as Antistatic Threads

BASF CORP. *U.S. Patent* 4,835,056

An electrically conductive filament consists of a metal core of Pd, Au, Ag, Co, Ni, Cu, Sn, Zn or Cr, especially Ni plus a Pd catalyst, surrounded by a sheath of non-conductive polymer (polyacrylonitrile), and has resistance of 50–10 billion Ω/cm. The filaments have increased durability because the metal is impregnated into the polymer structure, and are useful as antistatic filaments for textiles, spark plug wires, and so on.

Conductive Plastic Articles for Static Reduction

CELANESE ENG. RESINS *U.S. Patent* 4,851,081

Conductive plastic articles are produced by etching plastic pellets, treating for 2 or 3 minutes with an aqueous Pd-Sn catalyst solution having 1.2–2.5 g/l $PdCl_2$, 80–150 g/l $SnCl_2$ and 280–360 g/l HCl, electroless coating with 15–25 wt.% Ag, Ni or Cu, and heating the coated plastic. The conductive plastic is used to reduce static in medical instruments, conveyor belts, video and audio parts such as hubs.

Thin Film Magnetic Heads with Magnetic Films Containing Palladium

HITACHI K.K. *Japanese Appl.* 1/124,108

Thin film magnetic heads for high density recording have a magnetic substrate with a pair of magnetic films arranged on the substrate facing each other. The magnetic films consist of a quaternary amorphous alloy containing 0.2–6 at.% Pd, 3.5–7 at.% Hf, 1–4 at.% Ta, and Co, and show improved corrosion resistance, high saturation magnetic flux density, and low magnetostrictive constants.

Electrochromic Device for Numerical Display

CANON K.K. *Japanese Appl.* 1/136,129

An electrochromic device is prepared by providing transparent electrodes on both faces of a substrate, and then forming Ir oxide films simultaneously on both electrodes by anodic oxidation. An Ir oxide film is produced with a large change in colouring and discolouring concentration with one process.

Ruthenium Metal for Lead Switch Contact Material

TANAKA KIKINZOKU KOGYO *Japanese Appl.* 1/147,058

Vacuum-evaporating Ru metal for lead switch contact material uses a more than 99.98% Ru metal containing ppm amounts of Cd, As, Na, K, Zn, Mg, Ca, Sr, Bi, Sb, Ba, Pb, Al, Si, In, Mn, Ag and Sn.

Material for Photomagnetic Recording Medium

NIPPON TELEG. & TELEPH. *Japanese Appl.* 1/150,259

A magneto-optical thin film material contains Pt, Mn, Sb, oxygen, and one or more of Pd, Rh, Ir, Au, Co, Cu and Sn. The material is used for a photomagnetic recording medium for high density and erasable photomagnetic recording, having large Kerr rotation angles, excellent oxidation resistance, lower Curie temperature than that of a PtMnSbO thin film, stability in air, and high S:N ratio.

Stable Oxide Superconductive Material

TANAKA KIKINZOKU KOGYO *Japanese Appl.* 1/153,535

A new oxide superconductive material which is stable and can be produced on a large scale contains at least one of Y, La, Pr, Nd, Sm, Gd, Tb, Dy, Ho, Er and Yb, at least one of Ba, Sr and Cr, and at least one of Ir, Os, Ru and Cu, excluding Cu alone. The material is produced by preparing an alloy and oxidising; the alloy being produced easily in a specific shape.

Radiation Electrode Material for Static Charging

MITSUBISHI METAL K.K. *Japanese Appl.* 1/164,971

An electrode material is made by implanting Pt ions on the surface of high melting point metals or metal alloy wire material, to give a Pt-ion implanted alloy layer. The electrode material is used for charging the photosensitive body of a copying machine, having a maintenance free, long service life.

Cladding with Silver-Palladium Alloy Inlay for Use in Electronics

TANAKA KIKINZOKU KOGYO *Japanese Appl.* 1/178,391

A cladding consisting of 30–60% of an Fe-Ni alloy base material and a Ag-Pd alloy (with 20% or more Pd) incorporated like an inlay is manufactured by continuously cladding the Ag-Pd alloy on the base material in single or multiple longitudinal belts, using the hot rolling adhesion method. The process provides an inlay cladding for use as contacts in electronics, for example of reed switches and keyboards.

High Purity Metals for Vapour Deposition

TANAKA KIKINZOKU KOGYO *Japanese Appls.* 1/180,960–64

Pt, Pd, Rh, Ir and Os metals having at least 99.98 wt.% purity contain by weight a total of 30 ppm of elements including Cd, As, Mg, Sr, Ca, Bi, Al, In, Ag, Si, Sn and so on. The metals are used for contact point materials for reed switching devices, giving a smooth film, and preventing boiling of the metal and splashing during vapour deposition caused by impurities.

Cobalt-Palladium Magnetic Layer for Recording Medium

SONY CORP. *Japanese Appl.* 1/191,318

A vertical magnetic recording medium has a Co-Pd magnetic alloy thin layer consisting of 10–40 at.% Co and 60–90 at.% Pd. The magnetic layer has high density and high vertical magnetic anisotropy, with magnetic properties equivalent to or higher than those of conventional Co-Cr alloy magnetic layers, and is used for high density recording.

Improved Magnetic Powder

TOSHIBA GLASS K.K. *Japanese Appl.* 1/200,605

A magnetic powder consisting of a hexagonal system ferrite such as Ba, Sr, Pb or Ca ferrite, contains 5–500 ppm of at least one element selected from Pd, Rh and Ru, and 5–100 ppm of Pt. The magnetic powder has a uniform and narrow distribution plate form ratio, and is suitable for production of a high density storage medium with a high S:N ratio.

Metallising Composition for Ceramics

NGK SPARK PLUG K.K. *Japanese Appl.* 1/201,090

A metallising composition consists of inorganic components having 100 parts wt. CuO-Cu with 30–70 wt.% CuO and 70–30 wt.% Cu, up to 10 parts wt. MnO_2, up to 7 parts wt. Pt and/or up to 5 parts wt. Au, and an organic binder and solvent.

Magnetic Head for Floppy Disc Drives

HITACHI K.K. *Japanese Appl.* 1/205,504

A magnetic head has a magnetic core material whose surface is a quaternary amorphous magnetic alloy containing Co, at least 5 wt.% Hf, at least 1 wt.% Ta, 10–13 wt.% Hf+Ta, and 0.5–4 wt.% Pd. The magnetic head is used for floppy disc drives and VTRs, and is capable of high density recording against a high coercive force medium.