NEW PATENTS

ELECTRODEPOSITION AND SURFACE COATINGS

Single-Phase Platinum Aluminide Thermal Barrier

GENERAL ELECTRIC CO *European Appl.* 1,273,681

A thin layer of Pt was applied to a substrate of a gas turbine component. After exposure to Al a single-phase diffusion Pt aluminide bond coat was formed, and the surface was grit blasted to a finish of ~ 32–63 Ra. This coating was oxidised at elevated temperature and partial O_2 pressure to form a thin Al_2O_3 scale, prior to coating with a YSZ top coat. The life of the thermal barrier coating system is improved.

Palladium Plating Solution

KOJIMA CHEMICALS CO LTD *World Appl.* 02/103,084

A Pd plating solution comprises a soluble Pd salt with (in g l^{-1}): 0.1–40.0 Pd, 0.01–10 of a pyridine carboxylic acid and 0.002–1.0 of at least one of soluble Fe, Zn, Tl and/or Te salts, 0.005–10 of amine derivative of pyridine carboxylic acid, an aldehydobenzoic acid derivative, and an anionic or amphoteric surfactant. The Pd films have high purity, stability and gloss, are of thickness ≥ 5 μm and free from cracks.

APPARATUS AND TECHNIQUE

Oxygen Sensors

PRESENS PREC. SENSING GmbH *World Appl.* 02/103,334

A sensor uses microtitre plates with depressions for receiving samples to measure O_2. The depressions contain luminescent or fluorescent dyes of Pt, Pd or Ru complexes with phenanthroline, porphyrin or pyridine ligands embedded in particles of a gas-permeable but H_2O-impermeable matrix of a polystyrene derivative or copolymer.

High Resolution Electron Projection

CALIFORNIA INST. TECHNOL. *U.S. Patent* 6,515,292

A photocathode electron projector that emits monochromatic electrons of energy within 2% of each other, comprises a patterned Au-Pd quartz mask attached to the cathode. When illuminated by UV light, the cathode emits electrons. The UV is filtered to just above the work function of the Au-Pd material. The electrons meet parallel electric and magnetic fields which cause them to undergo cyclotronic orbiting. This avoids damage to the wafer, attached to the anode, that is being patterned.

Generation of High Purity Water Vapour

D. H. LORIMER *U.S. Patent* 6,524,934

A system for safer, continuous generation of ultra-pure H_2O comprises a catalyst vessel and several sorption vessels. O_2 and H_2 (together with an inert gas) are reacted with Pt or Pd in the catalyst vessel. The sorption vessels adsorb the H_2O vapour on molecular sieve material. When this is heated, ultra-pure H_2O vapour concentrations, ~ 100%, are produced.

HETEROGENEOUS CATALYSIS

Device for the Reduction of Nitrogen Protoxide

W C HERAEUS GmbH *European Appl.* 1,284,927

A device for reducing N_2O formed by the catalytic combustion of NH_3 and O_2 uses a catalyst system comprising a first and at least a second catalytic array. The first catalytic array is a Pt-Rh array and the upstream second catalyst array is a Pt-2–4 wt.% Rh array.

Redox Catalyst for Selective Catalytic Reduction

OMG AG CO KG *World Appl.* 02/100,520

A redox catalyst (1), for the selective catalytic reduction of NOx contained in the exhaust gas of diesel engines with the aid of NH_3, is claimed. (1) contains a catalytically active ingredient based on the $TiO_2/WO_3/MoO_3/V_2O_5/SiO_2/SO_3$ solid acid system and an oxidation catalyst based on Pt and Pd. The reduction catalyst is in the form of a cylindrical honeycomb catalyst with an inlet surface and an outlet surface. The oxidation catalyst is arranged on a section of the reduction catalyst next to the outlet surface. In (1), the catalytically active ingredient of the reduction catalyst acts as support material for the Pt group metals of the oxidation catalyst.

Hydrogenation of Aromatics

AKZO NOBEL NV *World Appl.* 02/102,939

A hydrocarbon feedstock, containing 10–80 vol.% of aromatics in the presence of a H_2-containing gas, is contacted with a hydrogenation catalyst comprising highly dispersed Pt and Pd, on a carrier. The catalyst comprises 0.25–1.0 wt.% each of Pt and Pd, calculated as metals. The carrier comprises SiO_2-Al_2O_3 with 5–50 wt.% of Al_2O_3 and 5–50 wt.% Al_2O_3 binder. The catalyst gives increased aromatics hydrogenation, as compared to a catalyst containing only Pt, or catalyst with > 50 wt.% Al_2O_3 binder, or catalyst containing > 50 wt.% Al_2O_3.

Carbon Nanotube Catalyst

INFINEON TECHNOL. AG *World Appl.* 03/004,155

A C nanotube catalyst material, a C nanotube arrangement and a method for producing a C nanotube arrangement are provided. The C nanotube catalyst material for catalysing the epitaxial growth of C nanotubes on the C nanotube catalyst material comprises Fe, and at least one additional material, selected from Pt, Pd, Cr, Ni or Co, and C.

Catalyst for Hydrogen Peroxide

ENI SpA *World Appl.* 03/014,014

A catalyst for the synthesis of H_2O_2 from H_2 and O_2 contains the Pt group metal(s) as active components, one or more polyolefins and a carrier. The H_2O_2 solution is for use in oxidation processes catalysed by Ti silicalite. The H_2O_2 process operates under high safety conditions with a high productivity and molar selectivity towards the formation of H_2O_2.

Hydromorphone and Hydrocodone Synthesis

ABBOTT LABORATORIES *U.S. Patent* 6,512,117

A ketone is produced from a narcotic alkaloid that has an allyl alcohol moiety by mixing the narcotic alkaloid with an acid in the presence of a catalyst, such as Pd black or activated Pd, in the substantial absence of H_2 gas. The method is useful for preparing hydromorphones and hydrocodones with novel impurity profiles. The narcotic alkaloid is selected from morphine, codeine or their salts.

Activation of Alkylaromatic Isomerisation Catalyst

UOP LLC *U.S. Patent* 6,512,155

The isomerisation of a non-equilibrium alkylaromatic feed of xylenes and ethylbenzenes involves contacting the feed with a catalyst while adding a trace quantity of H_2O to the reaction zone to lower the temperature. The catalyst comprises ~ 0.1–2 mass% of Pt group metal(s), an inorganic-oxide binder, and 5–90 mass% support selected from a mixture of a non-zeolitic molecular sieve and a pentasil zeolitic aluminosilicate. Equivalent isomerisation is effected at lower temperatures, so reducing losses and improving catalyst life.

Palladium-Immobilised Hydroxyapatite

ASAHI KASEI CORP *Japanese Appl.* 2002/275,116

A method of producing an aldehyde or ketone comprises oxidising an alcohol in the presence of a catalyst of Pd immobilised on the hydroxyapatite $Ca_{10-x}(HPO_4)_x(PO_4)_{6-x}(OH)_{2-x}{\cdot}nH_2O$ (where $0 \leq x \leq 1$ and n is 0–2.5.). The catalyst obtained is highly effective for oxidising alcohols.

Carboxylic Acid or Ester Production

DAICEL CHEM. IND. LTD *Japanese Appl.* 2002/275,128

A carboxylic acid or carboxylic ester is efficiently produced in a reaction between the corresponding compound that has a C–C unsaturated bond and CO gas in the presence of an oxidation catalyst composed of a Pd compound and a heteropolyacid or its salt (1). O_2 or a radical initiator are also present. The system may also contain an ionic compound containing a halide ion, and the preferable (1) to be used contains P and at least one element chosen from V, Mo and W.

HOMOGENEOUS CATALYSIS

Selective Isomerisation of α-Olefins

BP CORP NORTH AMERICA INC *European Appl.* 1,278,713

The isomerisation of α-olefins to internal olefins is performed selectively without significant concurrent isomerisation of vinylidene olefins, if present, in the reaction mixture. The catalysts employed in the process are Ru trihalides, preferably $RuCl_3$, $RuBr_3$, and/or their hydrated forms. When both vinylidene olefins and α-olefins are present in the feed or reaction mixture, only the α-olefins are substantially isomerised in the absence of isomerisation of the vinylidene olefins to trisubstituted olefins.

Hydroformylation of Unsaturated Compounds

BASF AG *European Appl.* 1,280,754

In hydroformylating ethylenically unsaturated compounds, especially internal branched olefins, at least one of them is reacted with CO and H_2 in the presence of a metal-ligand complex of Ru, Rh, Pd, Ir and/or Pt. The ligand, of general formula APR′, is monophosphine, monophosphinite or monophosphine amidite, and AP forms a 2-phosphatricyclo[3.3.1.1{3,7}]decyl radical. R′ is H or an organic radical with a molecular weight of up to 20,000 bonded with a C, O or N atom. Lower pressures and/or temperatures are required than with other P ligands.

Production of Thermoplastics

BASF AG *World Appl.* 03/011,941

Thermoplastic poly(3-hydroxyalkanoates) of molecular weight of > 6.000 g mol^{-1} and melting point 70–170°C are claimed. They are produced by reacting an oxirane compound with CO in the presence of a Rh(0), Pd(0), Ni(0), Fe(0) or Co(0) catalyst. The catalyst is chelated with neutral Lewis bases in the presence of diisocyanates, orthoesters, anhydrides, MgO, $MgCl_2$, $MgSO_4$, etc., at 1–250 bar and ≤ 150°C.

Chiral Organometallic Compounds

AVECIA LTD *World Appl.* 03/013,724

Chiral organometallic compounds (1), comprising non-symmetrically substituted cyclopentadiene complexed to a transition metal, are provided. The cyclopentadiene also has a second coordinating group which complexes the transition metal and is attached to the cyclopentadiene by means of a chiral connecting chain. Preferred transition metals include Pt, Pd, Rh, Ru, Ir, Co, Fe, etc. (1) find use in asymmetric synthesis to produce chiral compounds.

Metal-Chiral Phosphines for Asymmetric Reactions

PENN STATE RES. FOUND. *U.S. Patent* 6,521,769

Transition metal complexes of Pt, Pd, Rh, Ru, Ir, Cu, etc., based on chiral ligands, can be used in asymmetric catalysis. The chiral ligands include the chiral C1–C6 TunaPhos ligands; the Ru TunaPhos complex reduces ketones to the corresponding alcohols with 95–99.6% enantioselectivity. Asymmetric reactions include hydrogenation, hydride transfer, hydrosilylation, hydroboration, hydrovinylation, hydrocarboxylation, the Diels-Alder, aldol and Heck reactions, etc.

Polyolefins with Variable Density

MATERIA INC *U.S. Patent* 6,525,125

A polycyclic olefin composition with variable density comprises density modulators (1) dispersed in a polymer matrix. The matrix is prepared by the metathesis of an olefin monomer using a Ru or Os metal carbene catalyst, such as bis(tricyclohexylphosphine)benzylidene Ru dichloride, bis(tricyclohexylphosphine)dimethylvinylmethylidene Ru dichloride, etc. (1) are selected from metallic density modulators, such as W and W carbide, micro- or macroparticulate density modulators.

Oxidation of Amines

MURAHASHI SHUNICHI SUMITOMO CHEM. CO LTD *Japanese Appl.* 2002/265,430

Oxidation of amines comprises treating a primary or secondary amine with O_2 in the presence of a Ru catalyst, preferably $Ru_2(OAc)_4Cl$, to give a corresponding nitrile, imine or enamine. The method can be used on an industrial scale with high safety, and amines can be oxidised without differences between a chain amine and a cyclic amine, or between a primary and a secondary amine.

FUEL CELLS

Palladium Membrane Foil

BALLARD POWER SYSTEMS AG *U.S. Patent* 6,503,348

A Pd alloy membrane foil (1) for separating H_2 from the process gas mixture of a MeOH reformation system in a fuel cell-operated motor vehicle is claimed. (1) is produced by successive alternate galvanic depositions of metallic layers, from various baths, on the circumference of rotating deposition roller(s), wired as the cathode. (1) is removed from the roller(s) and tempered to convert the layer stack to a homogeneous alloy.

Single Room Type Solid Electrolyte Fuel Cell

NATL. INST. ADV. IND. TECHNOL. *Japanese Appl.* 2002/280,017

A single room type solid electrolyte fuel cell (1) has an anode of Sr-doped $Ln_{1-x}Sr_xCoO_{3-\delta}$, Ln = a rare earth, and a cathode containing Ni, CeO_2 and at least one of Pd, Pt, Rh, Ir and/or Ru. (1) can provide stable, high current in a gas mixture of CH_4 and O_2, even when operating at ≤ 600°C.

ELECTRICAL AND ELECTRONIC ENGINEERING

Semiconductor Devices

HITACHI LTD *World Appl.* 03/015,170

The O_2 barrier properties of a lower electrode of an FeRAM memory cell, the yield and the characteristics of the memory cell are improved by using a Pt film (1) as a lower electrode of a capacitor of an FeRAM memory cell. (1) is deposited on an Al-Ir alloy barrier layer, and is then annealed at 500–700°C in an O_2-containing atmosphere. Al_2O_3 forms at the grain boundaries of Pt crystals in (1). The PZP film serves as a capacitor insulating film.

Platinum-Rhodium Stack as an Oxygen Barrier

MICRON TECHNOL. INC *U.S. Patent* 6,524,867

A multiple layered electrically conductive film stack (1), which is a capacitor electrode and an O_2 barrier is claimed. It comprises alternate layers of a Pt-Rh alloy deposited by MOCVD in the presence of either oxidising gas, such as O_3, or reducing gas, such as H_2. (1) can be used in capacitors with high-k dielectrics. The electrode formed has a rough surface texture, which enhances memory cell capacitance.

High Voltage Termination

INTERNATIONAL RECTIFIER CORP *U.S. Patent* 6,525,389

A termination structure and reduced mask process, for a fast recovery epitaxial diode device or power semiconductor device, comprises at least two concentric diffusion guard rings and two spaced SiO_2 rings for an implant and drive system. A first metal ring overlies and contacts the outermost diffusion. Second and third metal rings act as field plates; the third is a continuation of the active area top contact and overlaps the second oxide ring. All rings are segments of a Pd or Al contact layer. The termination is for high voltage (~ 1200 V) devices.

Piezoelectric Thin Film

SONY CORP *Japanese Appl.* 2002/270,914

A piezoelectric thin film (1) is composed of a board, with a Pt thin film (2) and a Pb titanate zirconate thin film (3) formed on it, preferentially oriented to (111) and (100), respectively. Alternately, (2) is formed on the board as preferentially oriented to (111), with a first (3) of thickness 20–200 μm on it, and a second (3) after thermal treatment. (1) improves spontaneous polarisation characteristics and the freedom of design.

Gold-Palladium Alloy Powders

NORITAKE CO LTD *Japanese Appl.* 2002/275,502

Au-Pd alloy powder (1), used as conductive paste, has spherically shaped Au and Pd particles of average size 0.1–1 μm. (1) is made from a Pd and a Au solution; oxidising the Pd solution; mixing the Au and Pd solutions; forming a mist (2) of the resultant mixed solution; and then heating (2) for pulverisation.

MEDICAL USES

Platinum Coating of Titanium Cardiac Electrodes

MEDICO SpA *European Appl.* 1,284,148

A procedure for the Pt coating of Ti cardiac electrodes, comprises first spark erosion of the surface of the Ti electrode, followed by electrochemical deposition of Pt onto the electrode. The spark erosion is performed in a solution of MeOH and NaCl. After Pt electrodeposition the electrode is subjected to a flame to permanently fix the Pt to the Ti electrode.

Radiation and Thermal Energy Source

ATI MEDICAL INC *U.S. Patent* 6,497,647

Implantable seeds, designed to deliver thermal and ionising radiation to tumorous tissue, comprise rod-shaped ferromagnetic elements made of a Pd-Co alloy with a Curie temperature of ~ 40–100°C. These have Ti sleeving and Ti end caps that contain radioactive pellets. The ferromagnetic elements may also comprise rods with longitudinal channels in their outer surfaces, into which radioactive pellets are positioned. The radioactive pellets are ^{103}Pd or ^{125}I. The assembly is held together by an outer tubular sleeve or coating. Making the seeds and patient treatment are also described.