NEW PATENTS

METALS AND ALLOYS

Pt-Co Based Sputtering Targets

HERAEUS INC *World Appl.* 02/083,974

A Co-Cr-B-Pt sputtering target alloy having multiple phases and with enhanced product performance may also include Ta, Nb, C, Mo, Ti, V, W, Zr, Zn, Cu, Hf, O, Si or N. The alloy is prepared by mixing Pt powder with a Co-Cr-B master alloy, ball milling the powders and densifying the resultant alloy to form a magnetic sputtering target at pressures of ~ 15,000–30,000 psi, 1500–1900°F for 1 to 6 hours.

Colloid Solution of Metal Nanoparticles

POSTECH FOUNDATION *World Appl.* 02/087,749

A Pt, Pd, Ag, Cu and/or Ni nanoparticle colloid solution (1) and metal-polymer nanocomposites (2) are prepared by dissolving a salt of the metal and a H_2O-soluble polymer, such as polyvinyl pyrrolidone, in H_2O and an alcoholic solvent, purging with N_2 or Ar gas and radiating the solution with radioactive rays. (1) and (2) have uniform particle diameter and shape, and are used as antibacterial agents, conductive adhesives and inks, electromagnetic wave shields, etc.

PHOTOCONVERSION

Photocatalyst and Gas Deposition

SONY CORP *U.S. Patent* 6,471,929

A photocatalyst with superior durability has a fullerene polymer film (1) obtained by polymerising C_{60} or C_{70} fullerene molecules using electron beam, electromagnetic wave or electronic polymerisation. (1) is layered on a substrate and fine Pt and Pd particles (0.5 nm to 100 μm in size) are applied by sputtering, evaporation or coating. An apparatus to decompose gas includes a light source so that the gas is contacted with (1) under light illumination.

Photocatalyst Material

NIHON TETRA PAK KK *Japanese Appl.* 2002/186,860

A photocatalyst material (1) with excellent ability for decomposing pollutants comprises a composite film of TiO_2 and 3.0–80 wt.% Pd formed on the surface of a substrate by vacuum evaporation. The film is prepared by simultaneously evaporating TiO_2 and Pd by ion plating. (1) is highly hydrophilic and can decompose many types of organic substances.

Photocatalyst Manufacture

ISHIHARA SANGYO KAISHA LTD *Japanese Appl.* 2002/239,395

A photocatalyst (1) able to be excited by visible light irradiation is manufactured by adding a Pt halide compound to the surfaces of photocatalyst particles of TiO_2, etc. Particles of (1) and the Pt halide compound are then heated in a liquid medium. An accelerator containing hypophosphorous acid can be further added to the liquid medium at the time of heating. (1) is stable with high photocatalytic activity.

ELECTRODEPOSITION AND SURFACE COATINGS

Surface Coating of 'Black Platinum'

OMG AG CO KG *World Appl.* 02/095,088

A surface coating (1), 1 nm–10 μm thick, is formed from a fine dispersoid of modified Pt black particles, and comprises Pt and Si and/or a Si compound. An organic Pt(0) complex with 1,3-divinyl-1,1,3,3,-tetramethyldisiloxane, which can decompose at < 200°C, is applied to the surface of a substrate and is then thermally decomposed. (1) protects against mechanical, chemical and/or thermal effects, and can be used as an antiadhesion, antireflective or catalytic layer.

CVD Ruthenium Seed Layer and Ruthenium Thin Film

APPLIED MATERIALS INC *U.S. Patent* 6,479,100

A Ru seed layer is formed on a substrate by introducing a Ru-containing compound (1) and O_2 into a CVD apparatus and maintaining an O_2-rich environment for the initial formation of a Ru oxide seed layer. (1) is vaporised; and the Ru oxide seed layer (2) is deposited on the substrate by CVD. (2) is annealed in a gas ambient to form a Ru seed layer. A Ru thin metal film can also be deposited by MOCVD.

Electroless Platinum Plating Solution

TANAKA KIKINZOKU KOGYO KK *Japanese Appl.* 2002/173,780

An electroless Pt plating solution contains a hexaaminoplatinum complex (1). A salt of (1), which is used as the raw material, is made into an aqueous solution, and CO_2 is passed through to produce the carbonate of (1). This carbonate is then dissolved with acid. The plating solution enables continuous plating operation with extremely high stability, and further enables the production of a thin Pt film of high quality.

APPARATUS AND TECHNIQUE

Light-Emitting Devices

SRI INTERNATIONAL *World Appl.* 02/094,910

Conjugated polymers (1) with good solubility and semiconductivity which display high photoluminescent and electroluminescent efficiency are claimed. (1) contain a luminescent dopant of Ir, Os, Pt, W, Eu and Au complexes, with a bidentate or tetradentate ligand. Electroluminescent devices and other devices containing (1) are also provided.

Enzyme Electrode

SANKYO CO LTD *Japanese Appl.* 2002/189,012

A Pt electrode, which can be used as part of an enzyme electrode and is stable over time, comprises a Cu foil, a Ni layer, a Pd/Ni layer, and a Pt layer sequentially laminated on an insulating substrate. Superior accuracy in analysis and reproducibility are obtained. The Pt maintains very close contact and has high surface smoothness.

Sensor for Measuring Hydrocarbon Concentration

NATL. INST. ADV. IND. TECHNOL. *Japanese Appl.* 2002/202,281

A gas sensor to measure the concentration of a specific gas, such as a hydrocarbon in a gaseous mixture of CO, NO, H_2, etc., is claimed. It comprises a reference electrode (1) containing baked Pt paste formed on one surface of an O ion conductive solid electrolyte of ZrO_2-stabilised Y_2O_3, and a detection electrode (2) of 10 wt.% $SrCeO_3$ and 90 wt.% Pt formed on the other surface. The difference in electromotive force between both electrodes provides the measurement; (1) is exposed to air at 550–750°C while (2) is exposed to the gas to be measured.

HETEROGENEOUS CATALYSIS

Palladium Hydrogenolysis Catalyst

NE CHEMCAT CORP *European Appl.* 1,238,700

A hydrogenolysis catalyst with high hydrogenolysis performance at low temperatures comprises: (a) a component selected from Pd oxide, Pd oxide monohydrate and $Pd(OH)_2$ where Pd is in the divalent oxidation state, and (b) a component selected from Pt, Ru, Rh, Ir and Au, carried on a non-organic porous support. The Pd catalyst can be used for debenzylation, hydrodesulfurisation and dehalogenation reactions, and for the hydrogenolysis of esters.

Purifying Styrene Feedstock

FINA TECHNOLOGY *European Appl.* 1,256,559

A method to remove contaminants, such as phenylacetylene, from a crude styrene feedstock involves the catalytic reduction of phenylacetylene via injection of a reducing agent, such as H_2, to produce styrene. The reaction proceeds in the presence of a catalyst in the form of cylindrical pellets comprising < 0.3 wt.% Pd/Ca aluminate. Two reactor units, both containing the Pd/Ca aluminate catalyst, are used and output of phenylacetylene is reduced to < 10 ppm.

Preparation of 1-Methyl-3-phenyl-piperazine

NEULAND LABORATORIES LTD *World Appl.* 02/090,339

1-Methyl-3-phenyl-piperazine (1) is produced by mixing 1-benzyl-2-phenyl-piperazine first with formic acid solution, then with formaldehyde solution, and heating to 70–80°C. After treating with NaOH solution at < 25°C, filtration, washing and drying, 1-benzyl-4-methyl-2-phenyl-piperazine is obtained. Acetic acid is then added in the presence of Pt/C catalyst at a H_2 pressure of 3.5–4.0 kg cm^{-2}. After further treatments, (1) is obtained in an air oven.

Purification of Diesel Engine Exhaust Gas

KH CHEMICALS CO LTD *World Appl.* 02/092,224

A thermally and chemically durable catalyst for purification of diesel engine exhaust gas comprises 0.01–90 wt.% Pt, Pd, Rh, Ru and/or Re supported on a S-resistant refractory oxide, such as SiO_2, etc. A solid acid precursor and/or H_2SO_4 are also added. The catalyst is effective at removing particulate matter, hydrocarbons and NOx at low temperatures.

Direct Synthesis of Hydrogen Peroxide

ENI SpA *World Appls.* 02/092,501–502

H_2O_2 is produced from H_2 and O_2 in a reaction solvent that contains a halogenated promoter and/or an acid promoter, in the presence of a supported catalyst, such as 0.01–5 wt.% Pd and 0.01–1 wt.% Pt on activated C. The reaction solvent consists of alcohol(s), an aliphatic ether and optionally H_2O, and may contain 5C–32C hydrocarbons. The process operates under high safety conditions with a high productivity and molar selectivity towards the formation of H_2O_2.

Fuel Reformer

SUZUKI MOTOR CORP *Japanese Appl.* 2002/226,202

A fuel reformer capable of a shorter warm-up time to the start of operation of a reforming unit comprises a reforming part which generates H_2 gas for steam reforming in a reforming catalyst layer while feeding heat to MeOH. A combustion part supplies the heat generated by burning a fuel in the combustion catalyst layer to the reformer. The reforming and combustion layers and catalysts are on either side of a thin metal sheet. The combustion catalyst contains 0.5–5 wt.% Pt. Excellent reforming is obtained.

HOMOGENEOUS CATALYSIS

Suzuki-Miyaura Cross-Coupling

UNIV. NEW ORLEANS RES. *World Appl.* 02/072,511

A $Pd(OAc)_2$/diazabutadiene catalytic system for cross-coupling aryl halides with arylboronic acids is claimed. A combination of $Pd(OAc)_2$ and the diazabutadiene, 1,*N*,*N*′-dicyclohexyl-1,4-diazabutadiene, is an efficient catalyst for the Suzuki-Miyaura cross-coupling of various aryl bromides and activated aryl chlorides with arylboronic acids.

Water Soluble Palladium Complexes

COUNCIL SCI. IND. RES. *U.S. Patent* 6,469,169

A H_2O-soluble Pd complex is claimed for use as a catalyst in organic transformations, such as carbonylation, oxidation, hydrogenation, etc. The complex contains a phosphine ligand with three substituents selected from H, alkyl, arylalkyl and cycloaliphatic, at least one of which carries a sulfonic acid, and their salts. The Pd also carries a ligand of aryl or alkyl sulfonato; aryl or alkyl carboxylato; formato; or halide, such as Cl^-, Br^-, I^-. A further anionic chelating ligand consists of an N donor and an O^- group.

Acid Activation of Metathesis Catalysts

CALIFORNIA INST. TECHNOL. *U.S. Patent* 6,486,279

Highly active and stable Ru metal carbene complexes used as catalysts in olefin metathesis reactions have general formula $A_xL_yX_zRu{=}CHR'$ where $x = 0$, 1 or 2; $y = 0$, 1 or 2; and $z = 1$ or 2; R′ is H or a substituted or unsubstituted alkyl or aryl, L is any neutral electron donor, X is any anionic ligand, and A is a ligand of covalent structure connecting a neutral electron donor and an anionic ligand. Activation with HCl improves rates and yields of olefin metathesis reactions, such as ROMP, RCM, ADMET and cross-metathesis.

Production of Optically Active Alcohols

TORAY IND. INC *Japanese Appl.* 2002/155,096

A method to produce an optically active alcohol uses an optically active quadridentate Ru complex composed of an optically active compound containing two phosphines and two amide bonds in the same molecule and Ru, and asymmetric reduction of a carbonyl compound. An optically active amide is used as a source of asymmetry. The asymmetric reduction of a wide range of carbonyl groups, from aliphatic carbonyl compounds (a β-ketoester) to aromatic ketones, is claimed.

FUEL CELLS

Proton-Conducting Electrode

SONY CORP *European Appl.* 1,255,314

A proton conducting electrode (1) for a fuel battery comprises a mixture of an electron conducting catalyst (2) such as 1–50 wt.% Pt atoms, porous C powder fullerenes, C_m, where m = 36, 60, 70, 76, etc., (3) and a proton (H^+) dissociating group introduced into the C atoms. (1) is made by coating a mixture of (2) and (3) onto a gas transmitting current collector.

Anode Catalyst

OMG AG CO KG *European Appl.* 1,260,269

A Pt-Ru catalyst (1) for use as a fuel cell anode is prepared by suspending a support material in H_2O and heating to ≤ the boiling point. Solutions of H_2PtCl_6 and $RuCl_3$ are then added to the suspension, followed by addition of an alkaline solution. At pH 6.5–10, Pt and Ru are precipitated onto the support. Carboxylic acids and/or their salts are then added. (1) may optionally be calcined at 300–1000°C. In a fuel cell, (1) has high tolerance to CO poisoning.

Shift Converter with Improved Catalyst

UTC FUEL CELLS LLC *World Appl.* 02/090,247

A shift converter in the fuel processing subsystem of a fuel cell reduces the CO content in the process gas by a water gas shift reaction using a catalyst (1) selected from Pt, Pd, Rh and Au, preferably Pt. The Pt is supported by mixed metal oxides of CeO_2 and ZrO_2 in the ranges of 30–50 mol% and 70–50 mol%, respectively. Additional metal oxides may also be present. (1) obviates the need for prior reduction and minimises the need to protect the catalyst from O_2 during operation and/or shutdown.

Seawater/Acid/Catholyte Electrolyte

U.S. SECRETARY OF THE NAVY *U.S. Patent* 6,465,124

A Mg semi-fuel cell (1) has a Mg anode, a seawater/catholyte electrolyte (preferably containing acid to solubilise solid precipitates) and an electrocatalyst composed of Pd and Ir on C paper. The acid added to the electrolyte is preferably H_2SO_4, HCl, phosphoric acid, acetic acid or their mixtures. (1) provides a high energy density source for underwater vehicle applications with energy densities approaching 6–7 times that of Ag-Zn.

ELECTRICAL AND ELECTRONIC ENGINEERING

Contact Structure for Integrated Semiconductor

STMICROELECTRONICS SRL *World Appl.* 02/086,965

An integrated semiconductor device contains: a first conductive region (1); a second conductive region containing Pt (2); an insulating layer (3) between the two regions; and a contact structure (4) made of a conductive Ti- and TiN-layer. (4) coats the through opening in (3) and electrically connects (1) and (2). (4) is used in ferroelectric memory devices of the 'stacked' type, and is suited to the integration needs of the new CMOS technology.

Dielectric Composition with Reduced Resistance

E. I. DU PONT DE NEMOURS CO *World Appl.* 02/092,533

A dielectric composition (1) comprises a dielectric that is fireable in air and a conductive oxide selected from: Sb-doped Sn oxide, Sn-doped In oxide, a transition metal oxide with mixed valence states or which will form mixed valence states after firing in N_2 at 450–550°C, and conducting Pt group metal oxides, such as RuO_2. (1) has reduced electrical resistance and is used in electron field emission devices to avoid charging the dielectric near the electron emitter.

Composite Barrier Structure

SHARP LABORATORIES AMERICA INC *U.S. Patent* 6,479,304

An Ir combination film (1) used to make an electrode for a ferroelectric capacitor, also includes Ta and O. (1) effectively prevents O diffusion, and is resistant to high temperature annealing in O_2. When used with an underlying Ta or TaN layer, the resulting barrier suppresses Ir diffusion into any underlying Si substrate, so Ir silicide is not formed. (1) remains conductive, with no peeling or hillock formation.

Giant Magnetoresistive Stack

SEAGATE TECHNOLOGY LLC *U.S. Patent* 6,490,140

A giant magnetoresistive (GMR) stack for use in a magnetic read head includes a NiFeCr seed layer, a ferromagnetic free layer (1), a ferromagnetic pinned layer (2), a nonmagnetic spacer layer between (1) and (2), and a PtMnX pinning layer (3), where X is selected from Pd, Rh, Ru, Os, Cr, Nb, Re, Ta, Zr, Hf, Ni, Co and Fe. (1) has a rotatable magnetic moment and (2) has a fixed magnetic moment and is next to (3). A GMR read sensor has good thermal and magnetic stability and is used in magnetic data storage systems.

Perpendicular Magnetic Recording Medium

SAMSUNG ELECTRONICS KK *Japanese Appl.* 2002/216,341

A perpendicular reinforcement layer (1) which enhances perpendicular orientation between a substrate and a perpendicular magnetic recording (PMR) layer, is laminated in thicknesses of ≥ 15 nm. (1) contains Pt, Pd, Au, or their alloy. A base Ti layer may be placed between the substrate and (1). The effect caused by the difference in the diameter of the crystal lattice between the base layer and PMR layer is relaxed, while perpendicular orientation is enhanced.