

NEW PATENTS

ELECTRODEPOSITION AND SURFACE COATINGS

Film Deposition on Nanometre Structures

IBM CORP *U.S. Appl. 2002/0,090,458*

Thin film is deposited on a nanometre structure without filling holes and trenches by coating with a aerogel material and a metallic seed layer, such as Pt or Pd acetylacetonate. The coating is combined with a supercritical fluid, such as sc-CO₂, and a co-solvent, such as an alcohol. When the supercritical fluid is removed the coating solidifies into the thin solid film.

Chemical Vapour Deposition of Ruthenium Films

APPLIED MATERIALS INC *U.S. Patent 6,440,495*

A method to deposit Ru films via liquid source CVD uses vaporised bis(ethylcyclopentadienyl)Ru as the CVD source material gas at 100–300°C in a reaction chamber. An O₂ source reactant gas is provided. The substrate comprises Ti nitride, TiAl nitride or Ta pentoxide at a temperature of ~ 100–500°C and has a seed layer of Ru, Ir, Pt, Ru oxide, Ir oxide, etc., on which the Ru films are formed. The Ru film can be used as an electrode in a MIM capacitor.

APPARATUS AND TECHNIQUE

Thin Film Oxygen Sensor

PANAMETRICS INC *World Appl. 02/42,756*

An O₂ sensor operating at 300–350°C, comprises a crystalline ZrO₂ (1) sheet, and two porous Pt electrodes poisoned by Pb to a level that will inhibit cross sensitivity to reactive components, such as H₂. The Pt electrodes are arranged to induce superionic O transport along current paths in (1) at the electrode surface. O₂ concentrations of ppb can be detected.

Optical Switching Device

U.S. PHILIPS CORP *U.S. Appl. 2002/0,089,732*

An optical switching device comprises a transparent substrate and a switching film of a hydride of Sc and Mg, and optionally Ni, Al, Cr, etc., covered with a Pd or Pt catalytically active layer, in contact with an electrolyte. When a potential or current is applied between two electrodes, a change in optical transmission is detectable. The hydride is electrochemically switched from a low-H, mirror-like composition to a high-H, transparent composition, and vice versa, by H exchange. The device can be used in an optical switching element or sunroof.

Electrochemical Light-Emitting Elements

SHOWA DENKO KK *Japanese Appl. 2002/075,001*

An electrochemical light-emitting element (1) uses a Ru complex for its light-emitting layer together with a high-polymer solid electrolyte and an electrolyte salt. (1) has a high performance and needs only a low driving voltage to produce high light emission. (1) has superior stability, reliability, and low manufacturing costs.

Colouring Mater Sensitisation Type Solar Battery Cell

DAINIPPON PRINTING *Japanese Appl. 2002/093,475*

A colouring matter sensitisation-type solar battery cell is made from laminations of a transparent substrate, a transparent electrode layer, a power generation layer, a back electrode layer and a back substrate. The back substrate is pattern-coated with a Pt paste to form the back electrode layer, then baked with a coating liquid of fine TiO₂ grains to form an oxide semiconductor film. The film is impregnated, dried, and carries a Ru complex pigment sensitizer. Highly efficient power generation is obtained.

Hydrogen Separating Membrane

MITSUBISHI KAKOKI K. *Japanese Appl. 2002/119,834*

The manufacture of a highly permeable H₂ separating membrane (1) for separating H₂ from a H₂-containing gas is claimed. (1) is made by forming a Pd-based thin film on the surface of a porous carrier, and then depositing a Pd alloy or a metal to be alloyed with Pd on the pinhole parts of the membrane. After heat treatment, when a Pd-metal alloy is formed, the pinholes are effectively closed, and H₂ yield is increased. (1) is easily made.

HETEROGENEOUS CATALYSIS

Ruthenium Perovskite Production

NATL. INST. MATER. SCI. *European Appl. 1,233,002*

Ru perovskites of the type LaRuO₃ (1) are produced by reacting an aqueous solution of La and Ru ions with a precipitate-forming liquid to coprecipitate hydroxides of La and Ru which are then heat treated. (1) may also be precipitated onto a carrier from a homogeneous solution containing La, Ru and urea. The coprecipitated hydroxides have uniform dispersion and the resulting materials are efficient catalysts.

Destruction of CO, VOC and Organic Emissions

DEGUSSA AG *World Appl. 02/34,371*

A high performance catalyst (1) for the destruction of gaseous CO, VOC and halogenated organic emissions comprises a layer of Pt group metal deposited on an inert support. A washcoat into which the Pt is deposited consists of Al₂O₃ stabilised with La₂O₃, CeO₂ stabilised with ZrO₂ and Pr₆O₁₁. (1) is promoted by S-containing compounds selected from PtSO₄, H₂SO₄, (NH₄)₂SO₄, TiOSO₄, Ti₂(SO₄)₃, etc.

Production of High Quality Oil Bases

INST. FRANCAIS DU PETROL *World Appls. 02/48,289–290*

The simultaneous production of very high quality oil bases and middle distillates comprises successive steps of hydroisomerisation (1) and catalytic dewaxing (2). (1) is performed in the presence of a Pt group metal catalyst deposited on an amorphous acid SiO₂-Al₂O₃ support, with metal dispersion of ~ 20–100%. (2) occurs in the presence of a Pt or Pd catalyst and a molecular sieve selected from ZBM-30, etc.

Catalytic Converter for a Lean-Burn Engine

JOHNSON MATTHEY PLC U.S. Patent 6,413,483

A catalytic converter (1) for a lean-burn engine comprises a supported two-layer catalyst. The first layer contains Pt, K and a Ba NO_x storage component on a washcoat of a mixture of at least two of Al₂O₃, CeO₂ and/or ZrO₂. The second layer contains Rh on a washcoat of CeO₂ and ZrO₂. (1) further has an interlayer of a Ba compound on a washcoat. (1) is more selective for catalytic reaction between NO_x and/or nitrate with hydrocarbons and/or CO than for between hydrocarbons and/or CO with O₂. NO_x can be reduced to N₂ under constant lean to stoichiometric conditions without the need for rich spikes.

Hydrogenation of Acetylenes

UOP LLC U.S. Patent 6,417,419

Hydrogenation of 4C acetylenes in a liquid hydrocarbon stream that contains mainly butadiene is performed by contacting H₂ and the hydrocarbon stream with a catalytic composite on an inorganic oxide support. The catalytic composite has an average diameter of $\leq 800 \mu\text{m}$, with $\geq 70 \text{ wt.}\%$ of Cu and activator metal Pt, Pd, Ni, Co, Mn, or their mixture, being finally dispersed on the outer 200 μm layer of the support. The microsphere catalyst has much improved stability and selectivity compared to similar catalysts with particles of diameter $\sim 1600 \mu\text{m}$.

Vapour Phase Carbonylation with Iridium and Gold

EASTMAN CHEMICAL CO U.S. Patent 6,441,222

A vapour phase carbonylation process produces carboxylic acids and esters from a gaseous mixture of lower aliphatic alcohols, ethers, esters, CO and ester-alcohol mixtures using a solid supported catalyst. The gaseous mixture includes a halide promoter, and also H₂O and MeOH in a molar ratio of $\sim 0.01:1$ to $\sim 1:1$. The catalyst may be C, activated C, pumice, Al₂O₃, etc., containing 0.01–10 wt.% of Ir and Au each, preferably 0.1–2 wt.%. The catalyst also comprises another metal selected from alkaline metals, alkaline earth metals, Sn, etc. The carbonylation is performed at 100–350°C and a pressure of 1–50 bar absolute.

(S)-1-Phenylpropylamine

TOYO KASEI KOGYO Japanese Appl. 2002/088,031

(S)-1-Phenylpropylamine (1) is prepared by reacting (R)-1-phenylpropyl alcohol with diphenylphosphoryl azide as an azidation agent in the presence of a base to provide (S)-1-phenylpropyl azide (2). (2) is then subjected to a hydrogenating reaction in the presence of a Pd/C catalyst. (1) is produced in high quality and high yield.

Removing Carbon Monoxide

MITSUBISHI HEAVY IND. Japanese Appl. 2002/121,008

CO can be selectively reduced in a H₂-containing gas, to $\sim 10 \text{ ppm CO}$, by passing over a supported Ru metal catalyst at 60–350°C. Gas with an O₂:CO molar ratio of 0.01–0.5 is introduced to the catalyst. The difficulty of O₂ quality control is avoided.

HOMOGENEOUS CATALYSIS

C–C Coupling Reaction

DSM NV World Appl. 02/57,199

A C–C coupling reaction between an optionally substituted (hetero) aromatic bromide compound (1) and a second reactant, such as an aryl boric acid is claimed. The process was performed in the presence of an aprotic dipolar solvent, such as dimethylformamide or N-methylpyrrolidinone, a base and a Pd salt catalyst. The ratio between the quantity of Pd present in the Pd salt and (1) is 0.00001–0.1 mol%, preferably 0.01–0.1 mol%. (1) should contain at least one heteroatom chosen from N, O and S.

Acetic Acid and Methyl Acetate Production

ACETEX CHIMIE World Appl. 02/62,739

A continuous production of acetic acid and/or methyl acetate, based on carbonylation of MeOH, dimethylether, etc., is performed in a homogeneous liquid phase under CO. The catalytic system comprises Rh and a halogenated promoter, with H₂O at $> 14\%$ concentration. The process is gradually modified by adding an Ir compound. The system shifts from being a Rh-based homogeneous catalyst on its own to a catalyst based on Rh and Ir, or even Ir alone, without stopping the installation and reducing the H₂O content.

Ruthenium Alkylidene Catalysts for Olefin Metathesis

CALIFORNIA INST. TECHNOL. U.S. Patent 6,426,419

Ru alkylidene complexes (PCy₃)(L)Cl₂Ru(CHPh) (1), where L is a triazolylidene ligand, are claimed. (1) show high olefin metathesis activity, which is much higher at higher temperatures than that of the parent catalyst (PCy₃)₂Cl₂Ru(CHPh) (2). When L is 1,3,4-triphenyl-4,5-dihydro-1H-triazol-5-ylidene, (1) is able to catalyse the ring-closing metathesis of substituted dienes to give tetrasubstituted cyclic olefins in good yield. Additionally, (1) has a similar stability towards O₂ and moisture as that exhibited by (2).

Living Radical Polymerisation Initiator

KURARAY CO LTD Japanese Appl. 2002/080,523

A living radical polymerisation initiating system applicable to a wide range of radically polymerisable monomers comprises a halogenopentamethyl cyclopentadienyl bis(triarylphosphine) Ru, an α -halogenocarbonyl compound or α -halogenocarboxylic acid ester, and an amine. The system can easily and quickly produce a polymer with narrow molecular weight distribution while controlling the molecular weight.

Allene-Substituted Carboxylic Acid Ester

DENKI KAGAKU KOGYO Japanese Appl. 2002/088,026

A pure allene derivative free from substituent on the terminal and with a malonic acid ester (with a 1–8C straight chain alkyl, a branched alkyl with secondary or tertiary C, allyl, an aromatic hydrocarbon or butadienyl group) is produced using a Pd phosphine catalyst, and 2-chloro-1,3-butadiene. The diene is reacted with a Na compound of a malonic acid ester.

FUEL CELLS

Platinum-Ruthenium Electrocatalyst

NATL. INST. ADV. IND. TECHNOL.

Japanese Appl. 2002/075,384

Manufacture of an electrocatalyst (1) for an electrode catalyst joint body in a solid polymer fuel cell (SPFC) involves attaching a Pt-Ru catalyst layer to the surface of a polymer electrolyte membrane. (1) has superior oxidation activity for CO and alcohols. The SPFC has high performance.

Hydrogen Generating Device

MATSUSHITA ELECTRIC IND. *Japanese Appl.* 2002/121,006

H₂ is efficiently produced in a catalyst-containing H₂ generating device by suppressing catalyst deterioration due to S. The H₂ is produced by contacting a feed fuel, such as natural gas or LPG that might contain a S-based compound as an odorant, H₂O and air, with a Pt reforming catalyst. The catalyst also contains oxides of La, Ce, Al, Ga, Ti, Mg, Ca, Sr and/or Ba with Zr. The H₂ can be used in a fuel cell.

ELECTRICAL AND ELECTRONIC ENGINEERING

Surface-Metallised Pigmented Optical Body

3M INNOVATIVE PROPERTIES CO *World Appl.* 02/41,045

A colour-tailorable, surface-metallised, pigmented optical body comprises layered polymeric core(s) containing layer(s) of a thermoplastic polymer material. The thermoplastic polymer layers contain a dispersion of a particulate pigment such as C black, Fe oxides, etc. The metallic layer (1) comprises Pt, Ag, Au, Al, Cu and/or Ni, etc., at the outer surface(s) of the polymeric core (2). The transmission spectrum of the optical body differs from those of (1) and (2). The tinted polymeric films are used to provide neutral or coloured tint, in display devices, mirrors or other optical equipment.

Thermoelectric Device

IBM CORP

World Appl. 02/47,178

A thermoelectric device includes an electrical Pt conductor (1) thermally coupled to a cold plate and also a thermoelement electrically coupled to (1). The thermoelement has a plurality of tips to couple it electrically to (1). The tips provide a low resistive connection while minimising thermal conduction between (1) and the thermoelement. The device has improved efficiency and is used for cooling substances, such as integrated circuit chips.

Electroless Ni/Pd/Au Metallisation Structure

FLIP CHIP TECHNOLOGIES LLC *World Appl.* 02/58,144

A Ni/Pd/Au metallisation stack is formed upon the connection pads of integrated circuits at the wafer level by electroless plating. The interconnection pads can be Cu or Al (1). The lower Ni layer bonds securely to (1) and the intermediate Pd layer serves as an out-diffusion barrier for Ni. The upper Au layer can receive a variety of interconnect elements.

Ruthenium Oxide Film Formation

GENERAL ELECTRIC CO

U.S. Patent 6,417,062

RuO₂ films, for the fabrication of stable thin film resistors for microcircuits, are made by forming an inorganic Ru-based film (1) on a substrate, and then thermally decomposing a portion of (1) by exposure to high-intensity radiation, preferably visible light. RuCl₃·nH₂O and Ru(III) nitrosyl nitrate are used as the precursors. The method does not require thermal treatment which heats the bulk of the substrate, so can be used for non-ceramic substrates in printed circuit boards and flexible circuits.

Top Spin Valve Sensor

IBM CORP

U.S. Patent 6,437,950

A top spin valve sensor includes an IrMn pinning layer formed by ion beam sputter deposition. The magnetoresistive coefficient of the spin valve sensor is increased by placing an IrMnO seed layer between a free layer of the spin valve sensor and a first read gap layer of the read head. The free layer is preferably a NiFe-free film located between the first and second CoFe-free films.

Ferroelectric Capacitor with High Ferroelectricity

ROHM CO LTD

U.S. Patent 6,437,966

A ferroelectric capacitor, with maintained high ferroelectricity, comprises a Si substrate on which is a Si oxide layer, a lower electrode of an Ir-Pt alloy, a ferroelectric layer and an upper electrode. An Ir oxide layer is placed on the Si oxide layer, followed by an Ir layer on top, then the ferroelectric layer. The Ir-Pt alloy of the lower electrode can be formed to correspond to the ferroelectric layer. O vacancy in the ferroelectric layer can be prevented.

Electrically Conductive Antireflection Film

NIPPON ELECTRIC GLASS

Japanese Appl. 2002/071,906

An electrically conductive antireflection film (1) consisting of two layers is claimed. The first layer, of thickness 70–250 nm, contains at least one Pt group metal, Au and/or Ag, and their compounds, and a Co-containing inorganic pigment. The second layer has a refractive index of 1.3–1.6. (1) is coated on a glass panel of high light transmittance for use in a cathode ray tube. (1) reduces reflected light, enhances the contrast, and imparts superior antistatic performance and electromagnetic wave shielding.

MEDICAL USES

Microelectrode Catheter for Mapping and Ablation

C. R. BARD INC

World Appl. 02/47,569

A catheter (1) for mapping and/or ablation, includes a metallic cap of Pt or Au with a plurality of apertures and electrode(s) disposed in each aperture. Electrodes may be paired, or arranged along the length or circumference of the cap. (1) is used to treat a heart condition by placing it inside the heart and mapping a region of the heart with the mapping electrodes on the catheter or ablation using an ablation electrode disposed about the mapping electrodes.