

# NEW PATENTS

## ELECTROCHEMISTRY

### Cathode for Electrolysis

CSIR COUNCIL SCI. IND. RES. *U.S. Patent 5,855,751*  
A cathode for the electrolysis of an aqueous alkali chloride solution comprises a conductive steel substrate with a three component coating wherein: the first component is Ni oxide, the second is one or more of Rh, Ir, Pd and Os or their oxides, and the third is Au and/or Pt oxides, to reduce the H<sub>2</sub> overpotential. The electrode is used for non-mercury chloride cells, which currently use mild steel cathodes. The coating has catalytic activity and long term stability.

## ELECTRODEPOSITION AND SURFACE COATINGS

### Platinum Plating Bath

NIPPON ELECTROPLATING ENGINEERS K.K.  
*Japanese Appl. 10/330,986*  
The Pt plating bath contains a Pt compound consisting of a divalent H<sub>2</sub>O soluble Pt salt of organic acids chosen from saturated mono- or dicarboxylic acids, hydrocarboxylic acid or urea. The bath improves plating velocity and gives high current efficiency. Stability is enhanced by maintaining neutral pH.

## APPARATUS AND TECHNIQUE

### Gas Sensor for Automotive Applications

ARMINES ASSOC. RECH. DEV. METHODES  
*European Appl. 899,563A*  
A gas sensor for use in automotive applications, especially for measurements of CO, NOx and hydrocarbons, comprises a solid electrolyte of  $\beta$ -Al<sub>2</sub>O<sub>3</sub> upon which two electrodes, one made of Au and the other of Pt, are arranged to be exposed to the gas, a measurement device and a thermo-regulation system. The response of the sensor to the pollutants can be changed as a function of temperature, giving improved accuracy.

### Platinum Electrode Manufacture

GENERAL MOTORS CORP. *U.S. Patent 5,887,240*  
A Pt electrode for use in a vehicle exhaust gas sensor is made by mixing Pt-, ceramic- and C-powders to form an ink which is applied to a green body. The green body is then sintered to form the electrode. The electrode has high porosity and high current density, even when sintered at > 1500°C.

### Air Purifier

A. W. AISIN CO. LTD. *Japanese Appl. 10/296,082*  
A catalyst used as a filter for air purifiers consists of an optically active catalyst carrying 0.01–2.0 wt. parts of Pd as a photocatalyst for difficult-to-decompose volatile organic compounds (VOC). The VOC are quickly decomposed in the gaseous phase under optical irradiation at normal temperature. Energy consumption is reduced and catalytic activity is maintained.

## Powdered Solid Electrolyte

MITSUBISHI ELECTRIC CORP.  
*Japanese Appl. 10/328,286*

Powdered solid electrolyte used in an O<sub>2</sub> supply apparatus is mixed with specific catalysts to form an anodic catalyst layer attached to Pt-plated Ti metal and a cathode catalyst layer of 10–50% Pt/C. The current density of the film adhered between the anode and cathode is set to  $\leq 0.1$  A. The amount of O<sub>2</sub> supplied is adjustable and its generation is controlled. Deterioration of the film is prevented.

## HETEROGENEOUS CATALYSIS

### Automobile Diesel Engine Catalyst

MAZDA MOTOR CORP. *European Appl. 895,810A*  
An exhaust gas purifying catalyst for diesel engines comprises metal oxide particles containing Ce supporting dispersed Pd oxide particles. A Ce-containing coating layer covers the Pd oxide particles. Noble metals are fixed to the oxide by heat treatment. The catalyst gives enhanced purification and captures hydrocarbons in exhaust gas to crack and modify them to higher active hydrocarbons suitable as a reductant for NOx.

### Reduction of NOx

GENERAL MOTORS CORP. *European Appl. 899,002A*  
A catalyst for promoting the reduction of NOx in the exhaust of a fuel lean operating, hydrocarbon fuelled engine, comprises particles of a mixture of an alkali metal or alkaline earth metal ion exchanged ZSM-5 zeolite (SiO<sub>2</sub>:Al<sub>2</sub>O<sub>3</sub>  $\geq 50$ ) and Pt-Rh/Al<sub>2</sub>O<sub>3</sub> three-way catalyst. NOx is reduced by cyclically and alternately directing the exhaust stream between and through the above two catalyst beds, while cyclically and alternately injecting hydrocarbon into the beds.

### Rhodium-Tellurium Catalysts

MITSUBISHI CHEM. CORP. *European Appl. 904,836A*  
Production of unsaturated glycol diesters, such as 1,4-diacetoxy-2-butene and butenediol diesters, comprises reacting a conjugated diene with a carboxylic acid and O<sub>2</sub> in the presence of a solid Rh-Te (1) catalyst having the co-ordination number ratio (Rh-Te:Rh-Rh) in the EXAFS at the Rh-K edge of 50–99:1. (1) has high stability, activity and selectivity.

### Production of Shell Catalyst

DEGUSSA A.G. *European Appl. 904,838A*  
A shell catalyst, used for selective hydrogenation of C<sub>2</sub>H<sub>2</sub> in gas streams, has a non-porous inorganic support (BET surface area < 80 m<sup>2</sup> g<sup>-1</sup>) and is prepared by drying a suspension of H<sub>2</sub>O-soluble Ru, Rh, Pd, Os, Ir, Pt, Ag and/or Au compound(s) and a coating compound on the support and then activating the shell-coated support in a reducing gas stream. Simultaneous application of active and coating material is simple, giving a shell catalyst with an effective surface, suitable for use at high gas spatial velocities of > 3000 h<sup>-1</sup>.

## Production of 1,1,2,2,3-Pentafluoropropane

ALLIED-SIGNAL INC. U.S. Patent 5,856,594

Production of 1,1,2,2,3-pentafluoropropane (1) comprises contacting H<sub>2</sub> and 1,3-dichloro-1,1,2,2,3-pentafluoropropane over a catalyst supported on Al<sub>2</sub>O<sub>3</sub>. The catalyst is Pd, Pt, Ru, Rh and/or Ir, preferably Pd. The reaction is performed in the vapour phase at 185–260°C with a contact time of 10–25 s. (1) is used as a refrigerant, solvent, foam blowing agent or aerosol propellant. Very little reduction of the F atoms occurs.

## Hydrogenation of Hydroxyaldehyde

ARCO CHEM. TECHNOLOGY LP U.S. Patent 5,874,652

An aqueous mixture of 4-hydroxybutanal and 3-hydroxy-2-methylpropanal is hydrogenated in the presence of Pt-Ru/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> catalyst to produce 1,4-butanediol, 2-methyl-1,3-propanediol, < 0.01 moles of tetrahydrofuran per mole of 1,4-butanediol and < 0.1 moles of isobutyl alcohol per mole of 2-methyl-1,3-propanediol. The catalyst is used in a fixed bed process and has high activity and selectivity.

## Bimetallic Catalyst

FORD GLOBAL TECHNOLOGIES INC.

U.S. Patent 5,876,680

A bimetallic catalyst used for converting exhaust gases from a lean-burn engine, especially automotive engines, consists of a porous  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> support, and 0.1–0.5 wt.% W oxide and 0.5–4 wt.% Pt deposited on the support; the amount of metals being based on the weight of the support. The catalyst has excellent ability to reduce exhaust gases at lower temperatures than prior catalysts.

## Catalyst for Exhaust Gas Purification

NE CHEMCAT K.K. Japanese Appl. 11/28,359

The base structure of a catalyst for exhaust gas purification consists of a ferritic stainless steel containing in wt. %  $\leq 0.2$  C, 0.4–3.0 Si,  $\leq 2.0$  Mn, 13.0–20.0 Cr, 0.3–3.0 Mo, remainder Fe and impurities. The catalyst contains active ingredients chosen from Pt, Pd and Rh and a  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> carrier. A CeO<sub>2</sub> stabilised ZrO<sub>2</sub> is supported on the base. The catalyst has excellent corrosion resistance with high adhesion between the base and the catalyst.

## Decomposition of Nitrous Oxide

KYOCERA CORP. Japanese Appl. 11/33,401

A catalyst for decomposition of nitrous oxide in exhaust gases of I.C.E. comprises a spinel crystalline multiple oxide of 0.5–20.0 wt.% Pd, and Ga and Ni. About 5–75 wt.% CeO<sub>2</sub> is added to the multiple oxide. NO is decomposed into N<sub>2</sub> and O<sub>2</sub> without using a reducing agent, thus saving energy.

## Cyclohexanone Compounds

BAYER A.G. German Appl. 1/97/27,712

Cyclohexanone compounds (1) are prepared in consistent high yield by the catalytic hydrogenation of phenol compounds using Pd/C catalyst pre-mixed with a base component and H<sub>2</sub>O, at 100–250°C and 1–20 bar H<sub>2</sub> pressure. (1) include intermediates for pharmaceuticals and plant protectants and is obtained with high selectivity and in good reproducible yields.

## HOMOGENEOUS CATALYSIS

### Synthesis of Phenol

ENICHEM S.P.A.

European Appl. 894,783A

Phenol is produced by the catalytic oxidation of benzene and H<sub>2</sub>O<sub>2</sub> using Ti silicalite, with H<sub>2</sub>O<sub>2</sub> being prepared in situ by the reaction of O<sub>2</sub>, CO and H<sub>2</sub>O in the presence of: a Pd salt, a nitrogenated ligand and a non-co-ordinating organic acid. The process is simple and has sufficient activity and productivity to be used industrially. Phenol is used to prepare anti-oxidants, synthetic resins and insecticides.

### Production of Unsaturated Alcohols

MITSUBISHI CHEM. CORP. Japanese Appl. 10/298,124

Unsaturated alcohols are produced with high yields and selectivities by reacting conjugated alkydiene and H<sub>2</sub>O using a catalyst of a Pd compound and a phosphine. The skeleton of the unsaturated alcohol is obtained by the polymerisation. Of the unsaturated alcohols, octa-2,7-diene-1-ol and other octadienols are used as intermediates for *n*-octanol or its esters.

### Preparation of Aromatic Aldehydes

TOSOH CORP.

Japanese Appl. 10/330,307

Preparation of aromatic aldehydes (1) comprises reacting halogenated aromatic compounds with CO and H<sub>2</sub> in the presence of a catalyst comprising a Pd compound and tri-*tert*-butylphosphine and a base. (1) are prepared under milder conditions than previously.

### Production of DMUDO

DEGUSSA A.G.

German Appl. 1/97/30,546

Production of 6,10- and 6,9-dimethyl-5,10-undecadien-2-ones (DMUDO) comprises the telomerisation of isoprene with alkyl acetoacetates in the presence of a Pd compound, and a P component as the catalyst system (optionally in the presence of a protic additive). The resulting  $\beta$ -keto esters are then saponified and decarboxylated to produce DMUDO, with good, controlled selectivity, for Vitamin E synthesis.

### Rhodium-Based Hydroformylation Catalyst

HOECHST A.G.

German Appl. 1/97/40,672

A Rh-based hydroformylation catalyst contains Rh in elemental or bound form, polyethylene glycol, a P-containing ligand and H<sub>2</sub>O. The catalyst enables a high yield production of aldehydes by reacting 9–18C olefins with H<sub>2</sub> and CO. The organic phase and the aqueous catalyst phase can be rapidly separated, with no significant loss of Rh or ligands in the organic phase.

## FUEL CELLS

### Platinum Alloy for Fuel Cell Electrodes

EXXON RES. & ENG. CO.

European Appl. 899,348A

A C-supported, Pt-dispersed Zn alloy for fuel cell electrodes has 10–40 wt.% Pt particles, 20–30 Å in size, on a C support of surface area 100–500 m<sup>2</sup> g<sup>-1</sup>. A soluble Zn source is deposited on the Pt/C, and is calcinated and reduced to Zn. The alloy has good CO tolerance and can oxidise CO at low potentials.

## Phosphoric Acid Type Battery

TOSHIBA K.K. *Japanese Appl.* 10/334,928

The anode plate of a PAFC has a transition noble metal alloy catalyst layer and a Pt catalyst layer on corresponding sides of the oxidation-gas outflow and the inflow side of the reservoir. The PAFC reduces the current density distribution gap, the amount of phosphoric acid used and the phosphoric acid vapour on the oxidation-gas outflow side.

## Solid Polyelectrolyte in DMFC

JAPAN STORAGE BATTERY CO. LTD.  
*Japanese Appl.* 11/16,588

A polyelectrolyte used in DMFC consists of a double layered negative electrode catalysed layer. A Pt layer is formed on the inner side of the polymer electrolyte film. Another layer containing Pt and Ru is formed on its outer side. The amount of waste catalyst not participating in the reaction is reduced.

## Double-Layer Capacitor

SIEMENS A.G. *German Appl.* 1/97/24,712

An electrochemical double-layer capacitor (1) has a porous double-layer electrode made of C containing an electrolyte of poly(perfluoroalkyl)sulfone acid, and a porous H electrode made of Pt or a Pt alloy. The contact layers are C paper. (1) is used in combination with PEMFC for electrotraction. It has a high capacity in relation to volume and weight.

## ELECTRICAL AND ELECTRONIC ENGINEERING

### Piezoelectric Actuator

TOYOTA CHUO KENKYUSHO K.K.  
*Japanese Appl.* 10/273,361

A composition for a piezoelectric (PZT) actuator includes PZT ceramic with added precipitated Pt, Pd, Rh, Ru, Os, Ir or Au. Precipitation from solution occurs on the ceramic by heating and baking. Excellent displacement characteristics are obtained at lower cost, due to lower amounts of metal being used.

### Optical Recording Medium

KAO CORP. *Japanese Appl.* 11/25,515

An optical recording medium, such as for optical disks, has an optical reflex layer of Ag-Ru alloy, containing 0.5–20 at.% Ru laminated with a protective layer on a substrate. About 0.1–10 at.% of Rh, Pd, Ir or Pt are included in the alloy layer. The medium has a high reflecting rate and improved service life. The chemical stability of the optical layer is improved.

### Structure of a Semiconductor Electrode

FURUKAWA ELECTRIC CO. LTD.  
*Japanese Appl.* 11/26,396

A p-type compound semiconductor electrode has an Al layer partially formed on an insulating layer covering a Pt layer, and a Ti or Cr layer which are on the exposed surface of the semiconductor. Peeling of the electrode boundary surfaces and the insulating layer is prevented during wire or die bonding.

## Thick Film Conductor

SANKEN DENKI K.K. *Japanese Appl.* 11/31,872

A Ag-Pd thick film conducting paste for hybrid IC is printed and baked on a circuit board forming a thick conductor layer of 93.4–99.5 wt.% Ag and 0.5–6.6 wt.% Pd. A Ni layer is formed on the conductor layer by non-electrolytic plating and an Al wire is bonded to the Ni layer using ultrasonic oscillation. The connection strength is improved and embrittlement of the conductor layer is prevented.

## Perovskite Structured La-Sr-Ru Oxide

FUJITSU LTD. *Japanese Appl.* 11/53,935

A thin film electrode of perovskite crystal structure,  $\text{La}_x\text{Sr}_{1-x}\text{RuO}_3$ ,  $x = 0-1$ , is formed on a Si board by sputtering a target of  $\text{SrRuO}_3$  and La. It is used as an electrode of a ferroelectric random access memory. The film may be deposited on a Pt coated substrate.

## Sputtering Target Manufacture

TANAKA KIKINZOKU KOGYO K.K.  
*Japanese Appl.* 11/61,392

Ru for sputtering targets for the formation of Ru thin film is manufactured by melting Ru powder on a plate several times. A highly pure Ru ingot of > 99.9% purity is formed by cutting the plate. The Ru thin films are used in the manufacture of semiconductor devices. Bubbles of  $\text{H}_2$  and  $\text{O}_2$  gas formed on the film are removed together with impurities.

## Thin Film Magnetic Head

YAMAHA CORP. *Japanese Appl.* 11/66,514

A thin film magnetic head for a magnetic recording disk has a Pt spacer layer interposed in the multilayered magnetic core along the width direction of the magnetic layer. Magnetic field response occurs to high frequency, thus stratification becomes simple and complicated manufacture is avoided.

## Ruthenium-Doped Semiconductor

D. BIMBERG ET AL. *German Appl.* 1/97/47,996

Epitaxial production of Ru-doped semi-insulating III-V compound semiconductors is achieved using a Group V precursor having < 3 H bonded directly to the Group V element. The Ru layer is highly ohmic on both electron and hole injection and has low capacitance to avoid degradation of h.f. properties.

## MEDICAL USES

### Ceramic Material Composition

NIPPON HASHITOKURYOKU K.K.  
*Japanese Appl.* 11/1,738

Ceramic material for artificial teeth contains in wt.%: 19–20 Au, 19–30 Pd, 9–20 Cu, 0.02–0.03 Ir, 0.4–4.5 Sn, 0.5–4.5 Ga, 33–48 Ag, and impurities; the total amount of Sn and Ga is 2–5 wt.%. The alloy has excellent heat conductivity and improved chemical stability, and wear- and corrosion-resistance.

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