

# NEW PATENTS

## ELECTROCHEMISTRY

### Electrolytic Asymmetric Hydroxylation of Olefins

SEPRACOR INC. *World Appl.* 93/17,150A  
Electrolytic preparation of optically active compounds, such as glycols, more specifically enol ethers or vinyl halides comprises (a) asymmetric hydroxylation of olefins in a protic medium in the presence of a catalytic amount of an  $\text{OsO}_4$ -chiral ligand complex; and electrolytically regenerating  $\text{OsO}_4$ , for reuse in (a) from lower valent Os species. The process conditions give molar amounts of optically active compounds.

### Catalytic Activation of Cathode for Alkaline Electrolysis of Water

DEUT. AEROSPACE A.G. *German Appl.* 4,232,958  
Catalytic activation of a cathode for alkaline electrolysis of water involves adding a water soluble Pt metal salt to the electrolyte and electroplating the cathode with metal during cell operation. Activation reduces the over-voltage of the cathode by ~200 mV at 80°C, 240 mV at 120°C and 1 A/cm<sup>2</sup> and by 80 mV at 300 mA/cm<sup>2</sup> compared to a reversible H<sub>2</sub> electrode of a Pt cathode coated with Pt black. A dense deposit can be obtained, which remains active for months or years, and reactivation is simple.

## ELECTRODEPOSITION AND SURFACE COATINGS

### Electrodeposition of Uniformly Thick Metal Foil

TDK CORP. *European Appl.* 554,793A  
The electrodeposition of uniformly thick metal foil, especially Cu, on a rotating cathode includes using a concentric anode of multiple arcuate segments of valve metal coated with Pt group metal oxide and releasably secured to a back plate. Anode segments are easily replaced, giving simplified maintenance. The use of multiple segments minimises edge effects and contributes to improved uniformity of foil thickness with extended segment life.

### Diffusion Coating for Nickel and Cobalt Superalloy Turbine Engine Components

WALBAR INC. *European Appl.* 567,755A  
The high temperature oxidation and corrosion resistance of a metal superalloy is improved by applying a thin layer of a Pt group metal (preferably Pt) to the surface, heating to diffuse the metal into the surface and to form a Pt group metal aluminide, followed by packing the superalloy into a diffusion coating container, heating to diffuse Al and Cr into the surface, and removing the superalloy from the container and heating it to its solvus temperature. The resulting superalloy has a microstructure comprising an outer zone of Pt group metal aluminide.

### Electrolyte for Platinum Plating Bath

JOHNSON MATTHEY P.L.C. *World Appl.* 93/25,733A  
An electrolyte for a Pt plating bath comprises a Pt(II) salt present in solution as  $\text{Pt}(\text{H}_2\text{O})_4^{2+}$ . A process for electroplating a Pt or a Pt alloy film onto a conductive substrate using a plating bath is also claimed. The anion component of the salt is one or more groups or radicals derived from an organic or inorganic acid. The concentration of Pt in the bath is 0.005–0.0150 mol/dm<sup>3</sup>, and the operating temperature of the bath is preferably 15–60°C. The process operates at a current density of 0.03–10 A/dm<sup>2</sup> of substrate surface. The electrolyte allows the rapid deposition of high-quality, thick layers of Pt over a wide temperature range, even down to room temperature.

### Catalyst for Electroless Plating

HITACHI CHEM. CO. LTD. *Japanese Appl.* 5/255,861  
A catalyst for electroless plating is made by dipping carrier particles into an aqueous solution containing Pd ions, to precipitate Pd or Pd compounds on the surfaces of the carrier particles, and firing the particles in O<sub>2</sub>, to convert Pd into Pd oxide. The catalyst is used for making laminated boards for printed circuit boards.

### Platinum Plating Bath

NIPPON ELECTROPLATING ENG. K.K. *Japanese Appl.* 5/271,981  
A Pt alloy plating bath contains Pt as  $\text{Pt}(\text{OH})_6^{2-}$  complex ion and Sn, Zr or Pd. Sulphuric acid amide, Na- or K sulphuric acid amide is added to the bath, which is used at pH ≥ 11 and ≥ 60°C. The plating is carried out using a pulse power source. The Pt plating provides higher gloss and hardness than pure Pt. A glossy Pt-Zn alloy coating containing 96% Pt was obtained from a plating bath of K<sub>2</sub>Pt(OH)<sub>6</sub>, 10g Pt/l, ZnO alkali solution with 0.2 g Zn/l and 60 g KOH/l.

### Direct Formation of Electrocoating Layer on Non-Conductive Body

OKUNO PHARM. IND. K.K. *Japanese Appl.* 5/287,583  
A non-conductive body is coated with alkaline permanganate solution, then dipped into a catalytic solution containing Pd, Pt, Au, Ag or Cu, and thiourea, and a N-containing S compound consisting of a thiourea derivative; reduction-treating, activating, and forming the electrolytic layer on the body.

### Electroless Chemical Production

DAIMLER-BENZ A.G. *German Appl.* 4,220,621  
Electroless chemical production of a structured metal layer on a glass, quartz or ceramic substrate comprises forming a structured base layer containing SnO<sub>2</sub> on the substrate before deposition of a Pd layer. The substrate surface is treated with a reductant of Cl<sub>2</sub> or Cl-containing medium to convert SnO<sub>2</sub> into SnCl<sub>2</sub>. The base layer is produced as an In-Sn layer. The substrate is treated in a gas atmosphere, such as of Cl<sub>2</sub> or HCl vapour, or in a liquid bath, such as HCl.

## APPARATUS AND TECHNIQUE

### Optical Filter

PILKINGTON P.L.C.

*World Appl.* 93/24,849A

An optical filter used for absorption of electromagnetic (em) radiation in a narrow wavelength band comprises a transparent host material, such as a polymeric matrix of polycarbonate, and an em radiation absorbing material of 5,10,15,20-tetrakis (4-methoxyphenyl)-21H, 23H porphine Pd(II), and a narrow waveband em radiation absorber of vanadyl phthalocyanine. The filter absorbs em radiation of wavelength  $532 \times 10^{-9}$  m, while passing other wavelengths.

### Alcohol Sensor

FIGARO GIKEN K.K.

*Japanese Appl.* 5/302,905

A highly alcohol-sensitive sensor comprises a mixture of SnO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, and Pt in atomic ratio Sn:Y:Pt of 1:0.6–0.5:0.001–0.05. Preferably the amount of Y<sub>2</sub>O<sub>3</sub> to be added to SnO<sub>2</sub> is 10–50 mol.%, and the amount of Pt added is 0.2–3 mol.%. The alcohol sensor can detect an alcohol (EtOH) at the 10 ppm level and the interference caused by H<sub>2</sub>S or CH<sub>3</sub>SH is low, so alcohol exhaled can be detected.

## HETEROGENEOUS CATALYSIS

### Production of Dimethyl Naphthalene

MITSUBISHI GAS CHEM. CO. INC.

*European Appl.* 557,722A

Dimethyl naphthalene for use in the manufacture of 2,6-naphthalene dicarboxylic acid is produced by catalytic cyclisation dehydrogenation of 2-methyl-1-(*p*-tolyl)butene and/or 2-methyl-1-(*p*-tolyl)butane in the presence of 0.05–20 wt.% of a Pd catalyst such as PdCl<sub>2</sub>, Pd(NO<sub>3</sub>)<sub>2</sub>, Pd acetate, Na<sub>2</sub>PdCl<sub>4</sub>, H<sub>2</sub>PdCl<sub>4</sub>, etc., 0.1–20 wt.% of alkali and/or alkali earth metal compound(s), particularly LiCl, LiNO<sub>3</sub>, KCl, NaCl, NaNO<sub>3</sub>, and KNO<sub>3</sub>, on an Al oxide.

### Catalyst for Purification of Exhaust Gas

NIPPON SHOKUBAI CO. LTD.

*European Appl.* 558,159A

A catalyst for purifying exhaust gas comprises a monolithic honeycomb carrier with a first coating layer of CeO<sub>2</sub>, a first refractory inorganic oxide without noble metal and a secondary refractory inorganic oxide carrying 5–30 wt.% of Pt and/or Pd and/or refractory inorganic oxide with 1–20 wt.% of Rh; or a refractory inorganic oxide carrying 5–30 wt.% of Pt and/or Pd and 1–20 wt.% Rh. The catalyst has outstanding durability even under harsh conditions.

### Dehydrogenation of Saturated Hydrocarbons

INST. FRANCAIS DU PETROLE

*European Appl.* 559,509A

Dehydrogenation of a saturated aliphatic hydrocarbon charge comprising 3–5C per mole uses a supported catalyst of one or more Pt group metals, a promoter and an alkali or alkaline earth metal and with no H<sub>2</sub>. The preheated charge was introduced into one or more reactors for catalytic dehydrogenation. The process gives high conversion rates and high selectivity.

### Shell Catalyst Containing Palladium

HOECHST A.G.

*European Appl.* 565,952A

Shell catalysts, for gas phase production of vinyl acetate from C<sub>2</sub>H<sub>4</sub>, acetic acid and gas containing O<sub>2</sub>, are made by dissolving salts of Pd, K and Cd, Ba or Au in a suitable solvent, atomising the solution ultrasonically, impregnating the support one or more times and curing after each impregnation. The catalysts have better selectivity than those impregnated to the core.

### Palladium and Copper-Based Supported Catalyst

ITAL. MIN. UNIV. RICERCA SCI. TECHNOL.

*European Appl.* 567,198A

A Pd and Cu based supported metal catalyst, for selective hydrogenation of polyunsaturated hydrocarbons, is prepared by impregnating a porous support with a Pd and K salt solution, followed by treatment in a flowing H<sub>2</sub> stream; impregnating with a Cu salt solution and followed again by treatment with H<sub>2</sub>; and finally drying the resulting solid product. A typical composition is 0.05–0.4% Pd, 0.01–0.06% Cu and 0.01–3% K. The catalyst produces hydrocarbons containing the same number of C atoms but with a higher H:C ratio.

### Acetic Acid Production

CHIYODA CORP.

*European Appl.* 567,331A

Acetic acid production comprises introducing a feed including MeOH, CO, an alkyl iodide and a solvent into a reaction zone containing a solid Rh compound catalyst supported on an insoluble, pyridine-containing resin substrate, at 140–250°C and 15–60 kg/cm<sup>2</sup> pressure at a partial CO pressure of 7–30 kg/cm<sup>2</sup>. Acetic acid can be obtained in high yield and high selectivity even at relatively low reaction pressures. The high catalytic activity can be maintained for a long time.

### Catalyst for Selective Hydrogenation of Unsaturated Compounds

HUELS A.G.

*European Appl.* 576,828A

The catalyst, for use in the chemical and oil industries, contains Pd and/or PdO, with x = 0–1, on an Al<sub>2</sub>O<sub>3</sub> support, and may also contain 0.01–3 wt.% Pt, Ir, Ru and Rh (oxide). Fresh catalyst contains crystallites and has a Na content of 0.01–1%. The support contains η- and/or γ-Al<sub>2</sub>O<sub>3</sub>, and the noble metal is in an outer zone of thickness 5–200 μm. The catalyst is used to manufacture phenol and acetone. It has high initial selectivity on direct use, without time-consuming start-up procedures.

### Separating or Reducing Oxygen, Nitrite and/or Nitrate in Water

SOLVAY UMWELTCHEMIE G.m.b.H.

*World Appl.* 93/17,790A

The process comprises treating H<sub>2</sub>O with H<sub>2</sub> and contacting it with a catalyst of Pd and/or Rh or Pd and a metal of the Cu group impregnated in Al<sub>2</sub>O<sub>3</sub> carrier of θ and κ modification but with no or little α, γ and δ modifications. The pH is ≤ 8 and the process is repeated until the O<sub>2</sub>, nitrite and/or nitrate contents are zero or negligible. The catalyst is abrasion resistant.

## Removal of Sulphur and Nitrogen from Hydrocarbon Feedstocks

EXXON RES. & ENG. CO. *U.S. Patent 5,252,199*  
S and N are removed from hydrocarbon feedstock by treatment, in the presence of H<sub>2</sub>, with a highly dispersed multimetallic sulphide catalyst of MoS promoted with Pt, Pd, Rh or Ir in amount 0.1–10 wt.%, with the noble metal in an oxidation state > 0 and co-ordinated to S. The noble metal is preferably 0.25–5 wt. % Pt with Pt:Mo = 0.0025–0.05. The catalyst also contains Fe, Ni or Co sulphide in ratio sulphide:Mo = 0.1–0.5, and is prepared from a precursor complex ML<sub>2</sub> where M = Pt or Pd; or ML<sub>2</sub> where M = Rh or Ir, and L is dithiocarbamate, dithiophosphates, etc.

## Catalytic Production of Hydrogen Cyanide

E. I. DU PONT DE NEMOURS & CO. *U.S. Patent 5,262,145*  
The production of HCN is effected by feeding a gaseous mixture of N, O<sub>2</sub> and C compounds to a reactor containing two catalytic superposed piles both containing 33–67 wt. % of Pt group metal, either as mesh or particles. For gauze the mesh size of the first pile is finer than the second. The surface density of the gauzes decreases from the entrance to the exit according to the change in the O<sub>2</sub>:NH<sub>3</sub> ratio as the reaction proceeds. A catalyst pack for this reaction contains at least four gauze sheets.

## Allylic Chloride Production

DOW CHEM. CO. *U.S. Patent 5,262,575*  
Allylic chloride is produced by contacting an olefin-containing feed stream with a Pt- or Pd-chloride-containing chlorination composition, where the mole ratio of O:olefin is 0–1:20, at 200–300°C. The chlorination composition is separated from the feedstream and regenerated with Cl<sub>2</sub> in a second reactor before recycling for contact with the feedstream. High conversion of olefins and high selectivity to allylic products are achieved.

## Reforming Naphtha to Improve Octane Quality

EXXON RES. & ENG. CO. *U.S. Patent 5,269,907*  
The octane quality of a naphtha comprising a mixture of paraffins, aromatics and naphthenes is improved by reforming the feed over a high activity, high yield Sn modified Pt-Ir catalyst at reforming conditions sufficient to produce predominantly dehydrogenation and ring isomerisation reactions, yielding products useful as gasoline blending components. The catalyst comprises 0.1–0.7 wt. % Pt, 0.1–0.7 wt. % Ir and 0.02–0.4 wt. % Sn, with each metal being uniformly dispersed throughout an inorganic oxide support. A reforming unit is also claimed.

## Distillate Hydrogenation for Dearomatisation

AMOCO CORP. *U.S. Patent 5,271,828*  
Hydrocarbon feedstock, having a distillate portion with boiling point 150–700°F, is hydrogenated by contact with H<sub>2</sub> at 400–750°F and 300–2000 psig in the presence of 0.1–2.0 wt. % Pd and 0.1–2.0 wt. % Pt on a borosilicate support. The catalyst is durable and resilient with high crush strength.

## Catalyst for Decomposing Hydrazine

ISHIKAWAJIMA HARIMA HEAVY IND. *Japanese Appl. 5/261,285*

A catalyst to decompose hydrazine, used in a propulsion device for a space rocket, consists of a ceramic cordierite-type honeycomb with a layer of a gibbsite support loaded with Ir. A propulsion device is also claimed having a reaction part with the rear containing the catalyst and the front containing an Ir loaded granular hydrazine decomposition catalyst. Hydrazine is fed to the front catalyst and an exhaust nozzle jets out decomposition products at the rear.

## Catalyst for Nitrous Oxide Decomposition

SAKAI CHEM. IND. CO. LTD. *Japanese Appl. 5/317,648*  
The catalyst comprises at least one of Pt, Pd, Rh, Os, Ir, Ru or Re supported on a hydrophobic material. The catalyst contains preferably 0.3–2 wt. % of Rh or Ru. The hydrophobic material includes SiO<sub>2</sub> gel, activated Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>. The decomposition of N<sub>2</sub>O is performed at 300–500°C. The catalyst can decompose N<sub>2</sub>O contained in effluent from factories, refuse and sewage sludge incinerators.

## Low Pressure Hydrogenation of Aromatic to Cycloaliphatic Amines

BASF A.G. *German Appl. 4,207,314*  
Catalytic hydrogenation of aromatic amines to cycloaliphatic amines is effected at 120–250°C at 0.1–5 bar in the presence of NH<sub>3</sub>, using an alkali- and/or alkaline earth metal-doped catalyst containing supported Ru and Pd or Pt. Total amount of Ru + (Pd or Pt) is preferably 0.2–5 wt. % and the weight ratio Ru:(Pd or Pt) is preferably 0.2–5:1. This low pressure process overcomes catalyst deactivation by NH<sub>3</sub>, slow reaction and NH<sub>3</sub> condensation.

## HOMOGENEOUS CATALYSIS

### Production of Cinnamic Acid Derivatives

BAYER A.G. *European Appl. 564,919A*  
The production of cinnamic acid derivatives is effected by the reaction of bromo-aromatic compounds with acrylic acid derivatives in the presence of a Pd catalyst, an inorganic base, a phosphine, and an alcohol an/or a phase-transfer catalyst. The phosphine is used in large excess with respect to Pd. Cinnamic acid derivatives, such as 2-ethylhexyl and isoamyl *p*-methoxycinnamates, are useful as UV absorbers in cosmetics. The process gives high space-time yields without amine and solvent handling problems.

### Carbonylation of Olefinically or Acetylenically Unsaturated Hydrocarbons

SHELL INT. RES. Mij. BV. *European Appl. 565,199A*  
Carbonylation of an olefinically or acetylenically unsaturated hydrocarbon compound is effected by a reaction with CO and a hydroxy compound in a catalyst system of cationic Pd, a phosphine and a protonic acid; the reaction is carried out in the presence of a free radical inhibitor. The initial molar ratio of phenol:Pd is 5–30 and of protonic acid:phosphine is 0.5–10. The consumption of the phosphine is reduced.

## Production of Isoprene Derivatives

F. HOFFMANN LA ROCHE & CO. A.G.

*European Appl. 565,975A*

Production of di-, tri- or tetra-isoprene derivatives comprises asymmetric hydrogenation of an (E)- or (Z)-alkene or alkydiene in the presence of a Ru complex of an optically active atropisomeric diphosphine. The derivatives are intermediates for (R,R,R)- $\alpha$ -tocopherol, vitamin K1 and phytol manufacture.

## Stereospecific Production of Olefins

KURARAY CO. LTD.

*European Appl. 566,030A*

Production of olefins with an adjacent optically active C atom is carried out by reaction of Z or E allylic carbonate ester with formic acid or its salt in the presence of a tertiary phosphine and Pd salt catalyst. The olefin contains groups of 1-6C alkyl, H or 1-6C alkyl, H, OH or an organic group; or a pair of substituents on adjacent C atoms to form a bond, etc. The reaction proceeds stereospecifically to produce an optically active side chain to the ring system X in high yield and high selectivity. The process is industrially applicable and avoids the use of toxic materials.

## Asymmetric Hydrogenation Using an Optically Active Ruthenium Complex Catalyst

F. HOFFMANN LA ROCHE & CO. A.G.

*European Appl. 570,764A*

The preparation of 3-carboxy-perhydro-pyrazole, -pyridazine and 1,2-diazacycloheptane derivatives in (S) or (R) form comprises asymmetric hydrogenation of the corresponding 3-carboxy-3-pyrazoline, 3-carboxy-1,2,5,6-tetrahydro-pyridazine or 3-carboxy-1,2-diaza-3-cyclo-heptene derivatives or their salts in the presence of an optically active Ru diphosphine complex. The derivatives can be prepared with high optical purity.

## Preparation of Carbonyl Compounds

IDEMITSU KOSAN CO. LTD.

*European Appl. 572,036A*

Preparation of carbonyl compounds comprises reacting an olefin and O<sub>2</sub> or O<sub>2</sub>-containing gas in the presence of a Pd compound and/or metallic Pd, a polyoxoanion compound and an organic P compound in a solvent selected from O-, S-, N-containing organic compounds. The catalyst activity is well maintained and the carbonyl compounds are prepared with high efficiency and productivity. Deterioration in Pd catalyst activity caused by precipitation of Pd black is prevented.

## Palladium-Catalysed Alkylative Cyclisation

UNIV. LELAND STANFORD JNR.

*World Appl. 93/16,022A*

An alkylative cycloaddition process comprises reacting a compound RX (where R is vinyl, aryl or alkyl which does not have a  $\beta$ -H; X is bromide or iodide), with a 1,6- or 1,7-enzyme in the presence of a Pd catalyst to form a substituted bis(alkylidene)cycloalkane(s) or cyclohexadiene precursor as the reaction product. An E-bromoolefin is also claimed. The process is used in the production of vitamin D analogues, and is highly diastereoselective and simple.

## 3-Pentenoic Acid Preparation

E. I. DU PONT DE NEMOURS & CO.

*U.S. Patent 5,250,726*

3-Pentenoic acid is prepared from a reaction mixture of butadiene, CO, 1-20 wt.% H<sub>2</sub>O, a carboxylic acid solvent selected from 2-10C carboxylic acids, and benzoic acid optionally alkyl-substituted, where the total number of C in the alkyl group is  $\leq 3$ , a homogeneous Rh catalyst, a heterogeneous or homogeneous sulphonic acid catalyst, and an iodide promoter, at CO partial pressure of 50-1000 psi and 50-120°C. The rate of conversion of butadiene to 3-pentenoic acid at low temperature is increased.

## Three-Way Catalyst for Treatment of Exhaust Gas

ENGELHARD CORP.

*U.S. Patent 5,254,519*

A catalyst comprises a carrier with a catalytic coat of rare earth oxide-ZrO<sub>2</sub> support containing a 0.03-1.0 wt. % first Rh component, with respect to total weight of co-formed support, and a first activated Al<sub>2</sub>O<sub>3</sub> support with a 0.01-5 wt.% Pt component. The coat also contains a metal oxide, preferably Ni, Cu, Mn and/or Ge, to suppress H<sub>2</sub>S emission from the catalyst, a second Rh component dispersed on the first Al<sub>2</sub>O<sub>3</sub> support; and a second Al<sub>2</sub>O<sub>3</sub> support with a second Rh component. The rare earth oxide is Ce, Nb and/or Y.

## Regioselective Hydroformylation of Olefins

UNIV. NEW YORK STATE

*U.S. Patent 5,260,491*

Regioselective hydroformylation of olefins, where the olefin is reacted with H<sub>2</sub> and CO to yield two isomeric forms of an aldehyde, at least one being branched and predominating, is performed in the presence of cationic Rh bis-1,3,2-dioxa-phosphorus compounds as catalysts. The catalysts provide for the efficient, selective production of the branched chain aldehyde isomer, with  $\geq 75\%$ , preferably 100% regioselectivity. The aldehydes can be used to prepare ibuprofen and naproxen.

## Preparation of Aromatic Acetaldehydes

TAKASAGO PERFUM. CO. LTD.

*Japanese Appl. 5/229,981*

Aromatic acetaldehydes are prepared by oxidative decomposition of aromatic compounds with an allyl group such as propyl benzene, in the presence of Ru and phase transfer catalysts. The oxidising agent for the oxidative decomposition is a periodate. In an example RuCl<sub>4</sub> was used as the catalyst in the room temperature reaction. The reactions are safe and have excellent selectivity and yield for the compounds which are useful as synthetic intermediates to pharmaceuticals, agrochemicals and artificial sweeteners.

## Stereoselective Preparation of Cyclic Amines

KANEBO LTD.

*Japanese Appl. 5/255,285*

Optically active cyclic amines having benzyl, benzoyl, *p*-toluenesulphonyl or up to 10C alkyl, are prepared stereoselectively, in high yields, in an inert organic solvent in the presence of a Pd complex catalyst, in which an optically active phosphine is co-ordinated, and preferably in the presence of a base. The amine is a raw material for physiologically active substances.

## High Selectivity Hydrogenation of Acetylenes to Olefins

HENKEL KGAA.

*German Appl.* 4,211,126

Olefin production by  $H_2$  hydrogenation of acetylenic compounds is effected using a catalyst consisting of a Pd salt, an activator, and also a base to neutralise acid formed by reduction of the Pd salt; and/or  $H_2O$  and/or an inert  $H_2O$ -immiscible solvent. The catalysts give high conversion rates at 0–150°C and are effective in small amounts for use in tubular reactors.

## Production of Long Chain Methyl Alkyl Ethers

HENKEL KGAA.

*German Appl.* 4,221,099

Methyl alkyl ethers of formula  $R_1-O-CH_3$ , where  $R_1 = 6-22C$  aliphatic hydrocarbyl with 0–3 double bonds are produced by hydrogenation of  $R_1-O-CH_2R_1$  (1) in the presence of Pd catalysts and  $H_3PO_4$ . Hydrogenation is performed at 150–250°C and 10–100 bar, using 0.1–5 wt.% of Pd and 0.1–5%  $H_3PO_4$ , with respect to (1) and a Pd: $H_3PO_4$  ratio of 1:10–10:1. The ethers are raw materials for lubricants.

## FUEL CELLS

### Hydrogen and Hydrocarbon Gas Storage for Vehicle Fuel

BC LTD.

*World Appl.* 93/22,236A

Storing  $H_2$  or hydrocarbon gas comprises adsorbing or absorbing the gas with a transition metal dichalcogenide of formula:  $MX_2$  where  $M = Mo, W$  or  $Ti$ ; and  $X = S$  or  $Se$ ; preferably coated with Pt catalyst or H absorbent. A composition of formula:  $Y-MX_2$  is also new, comprising  $MX_2$  coated with Pt, Pd, Ni, Co, Fe, Mg, Zr, Cr, Al, Zn, Mn or their mixtures.

## CORROSION PROTECTION

### Composite Anticorrosive Material

LIMES K.K.

*Japanese Appl.* 5/311,402

Composite anticorrosive material, prepared with high energy efficiency at low cost, consists of a metal base with multiple Ni-Ta-Pt coatings containing Pt  $\geq 10$  at.% content in each layer. The layers are formed by ion beam sputtering or ion beam mixing. In an Ar ion beam sputtering unit a Ti sheet was coated with a 3 $\mu$ m layer of Ni-40at.%Ta-5at.%Pt, at an acceleration voltage of 3 keV, ion current 1.5 A,  $5 \times 10^{-6}$  Torr.

## ELECTRICAL AND ELECTRONIC ENGINEERING

### Magneto-Optical Layer and Magneto-Optical Recording Medium

TEIJIN LTD.

*European Appl.* 576,292A

A magneto-optical layer of a Co-Pt-Ru alloy has composition  $Co_aPt_bRu_c$  where  $a = 20-70$ ,  $b = 10-70$ ,  $c = 10-60$  and  $a + b + c = 100$ , the magneto-optical layer has an easy magnetisation axis perpendicular to the main surface of the magneto-optical layer. The magneto-optical layer has a magneto-optical effect at a wavelength shorter than currently used.

## Cobalt-Platinum Magnetic Film

EASTMAN KODAK CO.

*European Appl.* 576,376A

A CoPt magnetic film is deposited onto an underlayer deposited on a substrate with a curved surface. Also claimed is a method for depositing the magnetic film by sputtering an underlayer onto a substrate, and sputtering a CoPt film from a CoPt target onto the underlayer in an  $H_2$  and/or Xe atmosphere. The underlayer is preferably Cr or W. The magnetic film is  $> 300$  nm thick with a coercivity  $> 2300$  Oe. Film hardness is  $> 14$  G.Pa at a depth of 150 nm and a built density for the CoPt film of  $\geq 13$  g/cm<sup>3</sup>. The film is used for rotating cylindrical recording surfaces.

## Thick Film Resistor Compositions

E.I. DU PONT DE NEMOURS & CO.

*World Appl.* 93/23,855A

A thick film resistor composition comprises in wt.%: 5–30 of a Ru pyrochlore oxide, namely  $PbRuO_3$ , and 10–90 of a glass binder comprising a first glass containing  $PbO$ ,  $SiO_2$  and  $B_2O_3$ . The first glass comprises 5–30 wt.% of the resistor composition. The weight ratio of  $PbRuO_3$  to the first glass is 5:30–60:40. The thick film resistor composition minimises fluctuations and variations of resistance and has a small thermal coefficient of expansion.

## Palladium Thick Film Resistor

MOTOROLA INC.

*U.S. Patent* 5,250,358

A thick film resistor comprises a resistive sintered thick film bonded to a ceramic substrate of sintered Pd matrix, a first dispersed phase of BN and a second dispersed phase of  $Ta_2O_5$ . A preferred film comprises in wt.%: 1–15, preferably 2.5–7.5 BN and 2.5–7.5, preferably 4.0–5.0  $Ta_2O_5$ ; up to 2.5% Ca oxide borosilicate glass, up to 3% Ag and up to 5% alkaline earth titanate may also be present. The thick film resistor composition can be derived from a paste and is sintered by co-firing with a metal titanate substrate.

## Patterning of Conductive Metal Oxide Film on a Substrate

MOTOROLA INC.

*U.S. Patent* 5,254,217

Fabrication of a semiconductive device involves taking a substrate having a conductive metal oxide film selected from Ru, Rh, Ir or Os disposed on it and etching the oxide film using an  $O_2$  gas plasma. The Ru oxide has a stoichiometric composition of 2.03 atoms of O to every one atom of Ru. The  $RuO_x$  film is sputter deposited from a Ru target in an  $O_2$  ambient. This method is a means for plasma etching to provide a high resolution metal oxide pattern.

## Thick Film Resistor Paste

SUMITOMO METAL MINING CO.

*Japanese Appl.* 5/242,722

The paste comprises conductive particles coated with oxide(s) of Ru, Ir and Rh; a glass frit and an organic vehicle. The coating layer covers part or all of the surface of the particles. In an example,  $Re_2O_3$  powder,  $RuO_2$  and EtOH were mixed, filtered and dried to obtain surface modified powder. The paste, which is used for hybrid IC or thick film chip parts, is printed onto  $Al_2O_3$ , and sintered at a maximum of 850°C.

## High Density Recording Medium

TOSHIBA K.K. *Japanese Appl. 5/282,718*  
The medium comprises a laminate of dielectric film, a transition metal group-Pt type perpendicular magnetisation film and a metal reflecting film. When the dielectric film has a refractive index of  $n_1$  and attenuation factor  $k_1$ , and when the metal reflecting film has a refractive index of  $n_2$  and an attenuation factor of  $k_2$ , then:  $n_1 = 2.0-2.5$ ,  $k_1 \leq 0.3$ ;  $n_2 = 1.1-1.9$ ,  $k_2 = 1.6-2.5$ . The medium is used for high density recording and has an improved C:N ratio.

## Photomagnetic Recording Medium

SONY CORP. *Japanese Appl. 5/282,721*  
A photomagnetic recording medium comprises laminations of a polymer substrate, a dielectric layer, a Co-Pt type artificial lattice film (4 Å thick Co layers and 10 Å thick Pt layers were used in an example) and a UV ray curing type resin layer. The signal is preferably written by a laser of 3-6 mW power at rate of 1-3 m/s of linear speed. The medium has improved write/read characteristics for CD-MO use.

## Target for Corrosion-Resistant Magnetic Film

HITACHI LTD. *Japanese Appl. 5/290,330*  
A target for corrosion-resistant magnetic film comprises (Fe,Co)M(C,N) based alloy (where M is at least one selected from Hf, Zr, Ta, Nb, W, Mo and Ti) with added Cr and Rh and/or Ru. The target is used for depositing ferromagnetic film as a core material of a magnetic head.

## Magneto-Optical Recording Medium as Disk, Card or Tape

DAICEL CHEM. IND. LTD *Japanese Appl. 5/290,418*  
A magneto-optical recording medium has a recording layer based on Pt and Co, made of a composition-modulated multilayer film and an enhance layer made of substance(s) undergoing electron transition in the wavelength range of the laser beam for recording or regenerating information. The layers are laminated to form a single composite film. The enhance layer increases the Kerr rotation angle.

## Magneto-Optical Recording Medium with Fewer Regeneration Errors

SANYO ELECTRIC CO. *Japanese Appl. 5/290,419*  
A recording medium has a magnetic layer made of layers containing a noble metal(s) including Pt, Pd, Au, PtNi, PtFe and PtCo; and layers containing Fe, Co, Ni, FeCo and CoNi, laminated alternately, on a board as a recording layer. A UV-cured resin film is formed on the recording layer. Heat conduction is perpendicular to the recording layer.

## Magneto-Optic Recording Medium

SANYO ELECTRIC CO. *Japanese Appl. 5/298,759*  
A new magneto-optical recording medium consists of alternating laminate layer film of Pt and transition metal, a transparent board and a transparent underlayer which gives compact lamination of Pt sandwiched between them. The medium has improved crystallinity of Pt resulting in high coercive force of the alternating laminate layer film for high packing density recording.

## TEMPERATURE MEASUREMENT

### High Temperature Oxidation Resistant Noble Metal-Aluminium Alloy Thermocouple

NAT. AERONAUTICS SPACE ADMIN.

*U.S. Patent 5,275,670*

A thermocouple has an electropositive leg formed from a noble metal-Al alloy (such as Pt, Pd, Rh, Ir, Ag and Au) and an electronegative leg electrically joined at respective ends thereof to form a thermocouple junction. The electropositive leg comprises a Pt wire with a Pt-Al alloy coating and the electronegative leg comprises a Pt wire sometime coated with Pt-Al. The thermocouple provides accurate and reproducible measurement of high temperatures of 600-1300°C in inert, oxidising, or reducing environments.

## MEDICAL USES

### New Optically Pure Platinum Anticancer Complex

TANAKA KIKINZOKU KOGYO K.K.

*European Appl. 567,438A*

An optically active Pt complex compound is resolved by subjecting a solution of a d-isomer and of a l-isomer of a Pt complex compound to liquid chromatography and recovering the eluted solution of one of the two optically active-isomers. The new optically pure *cis*-oxalato (*trans*-1-1,2-cyclohexanediamine)Pt (II) has a melting point of 198.3-199.7°C.

### Anticancer Platinum Complex

SEIKAGAKU CORP.

*World Appl. 93/21,193A*

A new Pt complex contains a (3-8C cycloalkanediamine)Pt (II) with co-ordinated carboxyl and/or sulphate groups of a chondroitin-4-sulphate. The complex is electrophoretically homogeneous, with  $\leq 5$  wt.% Pt and H<sub>2</sub>O solubility at 20°C of  $\geq 10$  mg/ml with high selectivity for liver and spleen.

### Tetra Valent Platinum Diamine Antitumour Complexes

ROGER LAB BELLON S.A.

*World Appl. 93/23,410A*

New amino-aminomethyl-cyclic compound Pt(IV) complexes and their enantiomers, racemates, salts and hydrates contain optionally (i) unsaturated 7-12C polycyclic carbocyclic groups or (ii) saturated or partially unsaturated mono-, di- or tricyclic heterocycle containing 5-11 members and a heteroatom. The complexes are effective against colon and pancreatic adenocarcinoma in mice.

### Corrosion-Resistant Dental Alloy

C. HAFNER G.m.b.H. & CO. *German Appl. 4,211,403*

The dental alloy contains in wt. %: 85-95 Au, 2-12 Pt, 0-5 Pd, 0-5 Ag, 0.1-2 Sn and 0.05-0.5 Re. Preferably the alloy also contains 0.05-0.5% of each of Ir, Ru, Rh and Fe. The alloy is corrosion resistant and has an adequate melt-interval and an optimum coefficient of heat expansion.

The New Patents abstracts have been prepared from material published by Derwent Publications Limited.