efficients on models in the Boeing hypersonic wind tunnel.

Study of the Instability of Noble Metal Thermocouples in Vacuum

B. E. WALKER, C. T. EWING and R. R. MILLER, *Rev. Sci. Instrum.*, 1965, **36**, (5), 601–606

Studies were carried out at 800–1600°C on noble metal thermocouples and on thermoelements of Pt, Rh, Ir, Rh-Pt and Rh-Ir alloys. In vacuum, as in oxidising and neutral atmospheres, the main cause of instability is contamination by impurities, especially Fe, from the ceramic protection tubes.

Precise Temperature Measurement in Debye-Scherrer Specimens at Elevated Temperatures

R. G. MERRYMAN and C. P. KEMPTER, *J. Am. Ceram. Soc.*, 1965, **48**, (4), 202–205

In this method lattice parameters are measured against the temperature of a primary standard forming one leg of a calibrated thermocouple with both specimen and junction in the X-ray beam, and mixing the internal standard with the specimen so that the lattice parameters of the former denote temperature. Au is the primary standard, Pt is the other thermocouple leg and N.B.S. have calibrated the Pt : Au thermocouple at 0–1000°C

NEW PATENTS

METALS AND ALLOYS

Alloys

GENERAL DYNAMICS CORP. *U.S. Patent* 3,172,759

High temperature resistant alloys useful in the brazing of Nb and its alloys comprise 35–74 wt.% Cr, 25–65 wt.% Pd and 1–20 wt.% Ge.

High Strength Platinum-base Alloys

NEW ENGLAND MATERIALS LABORATORY INC. *U.S. Patent* 3,175,904

Dispersion strengthened precious metals and alloys are produced by providing under inert conditions Pt, Pd, Rh, Ir, or Ru or alloys of at least two of these metals with each other of such particle size that under normal conditions they would be pyrophoric, adding it slowly to a solution of refractory oxide-forming salt so that a thick slurry is formed and 6–20 vol.% refractory oxide will be provided, evaporating the solvent and drying the residue, spreading it evenly in a decomposition chamber and heating so that a precious metal powder product having a uniform dispersion of refractory oxide is formed.

ELECTROCHEMISTRY

Platinum-coated Titanium Anodes

IMPERIAL CHEMICAL INDUSTRIES LTD. *U.S. Patent* 3,177,131

Anodes are produced by applying to the Ti support a number of coatings of Pt-bearing preparation containing 2–9 wt.% Pt in the form of H_2PtCl_6 or H_2PtBr_6 or Pt resinate, in an organic vehicle, drying each coating and then firing it by heating in an oxidising atmosphere at 350–550°C to form a deposit consisting essentially of Pt.

Electrolytic Condenser Cathode

SIEMENS & HALSKE A.G. *German Patent* 1,190,105

A new form of cathode giving increased capacity consists of silver coated with Pt, Pd or Os.

ELECTRODEPOSITION AND SURFACE COATINGS

Deposition of Palladium

INTERNATIONAL NICKEL LTD. *British Patent* 994,560

A bath for the electroless deposition of Pd is operated at 68–100°C and comprises an aqueous solution of 1–20 g/l Pd (II), 0.04–05 g/l unsymmetrical dimethyl hydrazine, and one or more of the compounds NH_3, aminoethyl ethanolamine or amylamine in a total molar concentration equal to 100–350 g/l NH_3.

Electrodeposition of Platinum Group Metals

JAINOMOTO CO. INC. *British Patent* 998,709

Noble metal plated Ti electrodes are produced by immersing the Ti electrode in a fused bath containing alkali metal halide and dispersed graphite powder and then electrodepositing Pt, Pd, Rh or their mixtures on the roughened surface.

CATALYSIS

Production of Mesitylene

ESSO RESEARCH & ENGINEERING CO. *British Patent* 990,781

Mesitylene is produced by distilling a hydrocarbon mixture to provide a first fraction containing ethyl toluene, propyl benzene and mesitylene and isomerising the second fraction containing 1,2,4-trimethyl benzene by treating it with H_2 at 850–975°F, 200–350 p.s.i. and in the presence of an Al_2O_3-supported Cl_2-containing Pt catalyst.

Organopolysiloxanes

IMPERIAL CHEMICAL INDUSTRIES LTD. *British Patent* 990,800

Pt residues are removed from organopolysiloxanes prepared by a process involving the use of a Pt catalyst by treating the mixture in the presence of

an organic solvent and at 10–200°C with an alkali metal salt having an anion capable of co-ordination with Pt, and then separating the organopolysiloxane from the aqueous phase.

Reforming Catalyst

AMERICAN CYANAMIDE CO. *British Patent* 991,124

A reforming catalyst is produced by forming shaped γ-Al_2O_3 particles, immersing them in water at 35–90°C so that it percolates through the pore structure, separating said particles from it, calcining at 1000–1500°F, impregnating to provide 0.05–1 wt.% of a Pt group metal and then again calcining at 1000–1500°F.

Production of Hydrogen Peroxide

PITTSBURGH PLATE GLASS CO.
British Patent 991,338

A cyclic process for the production of H_2O_2 consists of using a working solution comprising a ketone, an aliphatic ester and hydrocarbon and carrying out the reduction step in the presence of Pd catalyst deposited on a porous siliceous material, which has been pretreated with an aqueous alkaline solution.

Production of Phenols

HALCON INTERNATIONAL INC.
British Patent 991,564

Phenol is produced by washing with alkali the crude mixture derived from oxidation of cyclohexane or alkylcyclo-hexane, vaporising the washed mixture, diluting it with 1–15 mol H_2 and contacting it at 250–425°C with a Pt/C dehydrogenation catalyst.

Catalytic Reduction of 3-Chloronitrobenzenes

GENERAL ANILINE & FILM CORP.
British Patent 991,789

Chloronitrobenzenes are reduced to the corresponding amino compounds by liquid phase hydrogenation at 25–125°C in the presence of a supported Pt group metal catalyst of at least 150 m^2/g surface area.

Trifluoromethyl Vinyl Ether

E. I. DU PONT DE NEMOURS & CO.
British Patent 992,659

Trifluoromethyl vinyl ether obtained by dehydrohalogenation of 2-chloro- or 2-bromo-ethyl trifluoromethyl ether may be polymerised in Pt tubes under usual reaction conditions.

Production of Aromatic Mononitro Compounds

GENERAL ANILINE & FILM CORP.
British Patent 994,706

Aromatic mononitro ethers are reduced to the corresponding amino compounds in the liquid phase and at 25–125°C by contacting them with hydrogen in the presence of a supported Pt gp. metal catalyst having at least 150 m^2/g surface area, e.g. 5% Pd/C catalyst.

Hydrogenative Cracking of Hydrocarbon Oils

SHELL INTERNATIONALE RESEARCH MIJ. N.V.
British Patent 995,174

Hydrogenative cracking is carried out at 268–454°C, 35–210 atm and in the presence of a catalyst consisting of a support comprising 50–90 wt.% SiO_2 and 10–50 wt.% Al_2O_3, MgO, ZrO_2 or B_2O_3, 0.1–5 wt.% Pt or Pt group metal and 0.1–5 wt.% F_2.

Catalysts

W. R. GRACE & CO. *British Patent* 995,596

Catalysts for use in the purification of exhaust gases from internal combustion engines comprise a support of at least 100 m^2/g surface area carrying 1–15 wt.% Co, 0.2–16 wt.% MnO_2 and 0.01–0.1 wt.% Pd and are produced by impregnating the support with a suitable solution, drying it and calcining for 1–16 h at 538–760°C.

Producing Hydrocarbon Mixtures

V.E.B. LEUNA-WERKE 'WALTER ULBRICHT'
British Patent 995,717

Hydrocarbons containing paraffins which boil above 350°C are subjected to catalytic hydrocracking, e.g. over Pt/Al_2O_3, and any aromatics removed to give a paraffin mixture.

Manufacture of B-Cyanethyl Silanes

WACKER-CHEMIE G.m.b.H. *British Patent* 995,863

The reaction of acrylonitrile and a silane is catalysed by a mixture of Pt and a nitrogenous cocatalyst.

Continuous Heat Treatment of Metal Strip

DAVY & UNITED ENGINEERING CO. LTD.
British Patent 995,883

In a new furnace, an inert atmosphere is maintained by passing the strip from an O_2-containing atmosphere into the furnace through an inlet provided with a catalyst, e.g. Pd/asbestos, which removes O_2 by reaction with a component of the furnace atmosphere, e.g. H_2.

Catalytic Hydrocracking

SOCONY MOBIL OIL CO. INC.
British Patent 996,302

A catalyst of unusual activity and selectivity for hydrocracking petroleum hydrocarbons consists of a catalyst metal, e.g. a Pt metal, on a crystalline aluminosilicate containing a rare earth.

Catalysts

W. R. GRACE & CO *British Patent* 996,844

Internal combustion engine exhaust gases may be catalytically burnt over a catalyst consisting of 4–16% CoO, 1–20% Cr_2O_3 and 0.01–0.1% Pd on a support.

Catalysts

W. R. GRACE & CO. *British Patent* 997,137

A catalyst for use in the oxidation of internal combustion exhaust gases comprises an Al_2O_3, SiO_2-Al_2O_3 or SiO_2-MgO support of at least 100 m^2/g surface area and carrying 5–20 wt.% CuO, 0.01–0.10 wt.% Pd and 1–15 wt.% Cr_2O_3.

N-Alkyl Hydroxylamines

E. I. DU PONT DE NEMOURS & CO

British Patent 997,300

N-Alkyl hydroxylamines are produced by hydrogenating a 1–3 C nitro-alkane in the presence of at least 1 equiv. H_2SO_4 at below 50°C and in the presence of Pt, Pd, Ru, Rh, Ir, Os or their oxides.

Hydrogenation Catalysts

UNION CARBIDE CORP. *British Patent* 997,614

An activated hydrogenation catalyst is produced by contacting a compound of Ru, Rh, Pd, Os, Ir or Pt with an Si-H bonded compound which contains at least one H atom bonded to Si in a sufficient amount to cause reduction of the valency state so that at least a portion of the metal is reduced to its lower oxidation state.

Metal Catalytic Igniter Members

ROLLS-ROYCE LTD. *British Patent* 998,455

The activity of a metal catalytic igniter member is improved by removing the surface layer by etching. After etching the member may be plated with a material (e.g. Pt) which will support catalytic ignition. The igniter is preferably a mesh and may be made of Pt or a Pt alloy, e.g. 20–30% Rh-Pt.

Preparation of Halogen-Containing Alcohols

SHELL INTERNATIONALE RESEARCH MIJ. N.V.

British Patent 998,892

Halogen-containing alcohol is obtained by contacting a halogen-containing carbonyl compound and hydrogen with 0.01–10 wt.% Ru supported on C, clay or Al_2O_3, at 15–350°C, 50–3000 p.s.i.g. and in an aqueous acidic hydrolytic medium containing at least an equimolecular amount of H_2O.

Olefine Oxidation Catalysts

FARBWERKE HOECHST A.G. *U.S. Patent* 3,172,913

Olefines are converted to aldehydes and ketones by contacting the hydrocarbon and oxygen with a neutral to acid liquid aqueous catalyst comprising (i) a Pd, Ir, Ru, Rh or Pt salt and (ii) a redox system while using two contact zones and regeneration of the catalyst system.

Hydrocracking Catalysts

UNION OIL CO. OF CALIFORNIA

U.S. Patent 3,173,853

High boiling hydrocarbons are subjected to hydrocracking by first freeing them of N-containing compounds and then contacting them at 500–850°F, 500-3000 p.s.i.g. and in the presence of 0.0075–1.5 wt.% steam with H_2 and a zeolitic Y crystal type molecular sieve supporting 0.05–2 wt.% Pt, Pd, Rh or Ir.

Reforming Catalysts

PULLMAN INC. *U.S. Patent* 3,173,856

Naphthas are reformed by contacting them at 600–1050°F and 100–750 p.s.i.g. in the presence of 0.5–20 moles H_2 with a fluidised catalyst comprising 75–99.9 wt.% ε-alumina and 0.1–25 wt.% Pt or Pd.

Reforming Catalysts

UNIVERSAL OIL PRODUCTS CO.

U.S. Patent 3,173,857

A catalyst for the hydro-reforming of hydrocarbons is produced by calcining a refractory metal oxide, preferably Al_2O_3, impregnating it with 0.01–2 wt.% Pt group metal, drying below 400°F, oxidising the composite at 400–600°F to decrease the volatile content below 5 wt.% combining halogen at any convenient stage and ensuring that the final catalyst contains 0.75–1.5 wt.% combined halogen.

Production of Dipyridyls

IMPERIAL CHEMICAL INDUSTRIES LTD.

U.S. Patent 3,173,920

2.2′-Dipyridyl and its alkyl derivatives are produced by heating pyridine or its alkyl derivatives at 250–450°C with 1–50 wt.% Al_2O_3 catalyst supporting up to 5 wt.% Rh, Os, or Ir.

Isomerisation Catalysts

SINCLAIR RESEARCH INC. *U.S. Patent* 3,175,014

Hydronaphthalenes are isomerised by contacting them at 400–900°F, 200–2,000 p.s.i.g. and in the presence of H with a catalyst comprising at least 75 wt.% activated Al_2O_3, 3–20 wt.% B_2O_3 and 0.01–2 wt.% Pt group metal, preferably Pt.

Esterification Catalysts

ESSO RESEARCH AND ENGINEERING CO.

U.S. Patent 3,176,038

Ethylenically unsaturated diesters are produced by contacting a mixture of 1 mol 4–25C unsubstituted α, β-mono-ethylenically unsaturated aliphatic carboxylic acid monoester and at least 2 mols 1–20C alkanol, with substantially hydrogen-free CO at 125–225°C, 500–3,000 p.s.i.g. and in the presence of 0.001–5 wt.% Rh_2O_3.

Reforming Catalysts

SOCONY MOBIL OIL CO. INC.

U.S. Patent 3,117,135

Hydrocarbon naphthas containing 0.07–0.7 wt.% S and at least 20 mol.% alkylcyclopentanes are reformed by passing them at 950–970°F and 100–750 p.s.i.g. in conjunction with H over a catalyst comprising Al_2O_3 supporting 0.1–2 wt.% Pt and 0.1–5 wt.% Cl. See also 3,177,136.

Cyclododecatriene Reduction

ESSO RESEARCH & ENGINEERING CO.
U.S. Patent 3,182,093

1,5,9-Cyclododecatriene is selectively reduced to 1,5-cyclododecadiene by means of a catalytic mixture of Ni and Pt at up to 200°C.

Removal of Acetylenes and Dienes from Propene

CHEMISCHE WERKE HULS A.G.
German Patent 1,190,457

Methyl acetylene and propadiene are removed from propene by selective hydrogenation over S-poisoned Pd/SiO_2.

Pyridine Production

FARBWERKE HOECHST A.G.
German Patent 1,192,648

Piperidine is catalytically dehydrated to C_5H_5N using SiO_2 containing only 0.02–0.2 wt.% Pt or Pd.

Catalyst for Polysiloxane Production

BUNDESREPUBLIK DEUTSCHLAND
German Patent 1,193,504

The reaction of H-terminated polyorganosiloxanes with allyl diketones to introduce terminal diketone groups is catalysed by a Pt metal.

Hydrogenation Catalyst

ESSO RESEARCH & ENGINEERING CO.
German Patent 1,193,630

Petroleum fractions boiling between 150 and 345°C and containing aromatics and S are improved by selective hydrogenation over a catalyst consisting of Pt/ε-Al_2O_3.

Production of Methyl Isobutyl Ketone

RHEINPRUESSEN A.G. *German Patent* 1,193,931

Methyl isobutyl ketone is produced from acetone and H in the presence of a mixture of acid cation exchanger and a selective hydrogenation catalyst, e.g. Pd/C.

Catalytic Olefine Oligomerisation

CONSORTIUM FÜR ELECTROCHEMISCHE INDUSTRIE G.m.b.H. *German Patent* 1,193,934

Olefines and acetylenes are oligormerised by water-soluble Pt metal salts, e.g. $PdCl_2$.

FUEL CELLS

Fuel Cells

THE ELECTRIC STORAGE BATTERY CO.
British Patent 991,928

A fuel cell has concentric, spaced electrodes and the O electrode is formed by a microporous cylinder of sintered Ni and Ag particles while the H electrode is made of sintered, finely divided Ag particles through which about 1 wt.% of finely divided PtO has been uniformly dispersed.

Fuel Cells

ESSO RESEARCH & ENGINEERING CO.
British Patent 993,662

A fuel cell comprises an electrolyte compartment containing an electrolyte comprising 3–50 wt.% H_2SO_4 and 0.1–1.0 wt.% HNO_3, electrodes formed of porous C impregnated with about 2 wt.% of a mixed metal catalyst containing 95 wt.% Pt, and 5 wt.% Au, an ion-permeable membrane separating the anode and cathode, means for passing fuel into dual contact with the anode and means for passing a fluid oxidant into the electrolyte compartment.

Fuel Cell Electrode

AIR PRODUCTS & CHEMICALS INC.
British Patent 995,151

A fuel cell electrode is produced by combining an aqueous solution of $PdCl_2$ or H_2PtCl_6 with activated C in 8–15 wt. excess, mixing the slurry to effect maximum adsorption, removing excess of liquid and drying the C slurry, using it to form a slurry and introducing it into the pores of thin C, Ni, Ag or Fe electrode matrix and drying said matrix.

Fuel Cells

LEESONA CORP. *British Patent* 995,903

A fuel electrode is used which is activated by a metal from the second or third transition series of Group VIII, e.g. Rh, Pd, or Pt.

Fuel Cells

LEESONA CORP. *British Patent* 998,925

A fuel cell electrode is formed by providing a highly porous thin plastic sheet coated with Ag, Cu, Ni, Cd, Pt, Au, or Pb or a sintered porous metallic matrix of Ag, Au, Pt, Cu, Cd, Al or Sn and then depositing on it a non-porous film of Pd or Pd alloy with 15–35 wt.% Ag.

Non-porous Electrodes

LEESONA CORP. *U.S. Patent* 3,180,762

An improved H diffusion concentration fuel cell has a non-porous Pd or Pd-Ag alloy membrane as the anode.

Fuel Cell Electrode

THE ELECTRIC STORAGE BATTERY CO.
U.S. Patent 3,181,973

An electrode consists of a powder mixture of thermoplastic resin, polyethylene oxide, a conductive material such as Ag, Cu or graphite and PdO which is compressed and shaped and then the PdO reduced to Pd in an alkaline electrolyte.

Fuel Cell Electrode

U.S. SECRETARY OF THE NAVY
U.S. Patent 3,183,122

A new electrode giving very rapid H molecule dissociation consists of a porous mass of Ni–Pd alloy containing 0.1–2% Pd.