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The Editor, Platinum Metals Review
The usefulness to industry of any one of the many metals and alloys from which a choice may nowadays be made depends naturally enough upon its providing some essential combination of properties that cannot be found elsewhere at comparable cost. The inherent characteristics of platinum—high melting point and exceptional resistance to corrosion combined with excellent working properties—opened up fields for its application from the very beginnings of modern industry and gave it, in fact, a unique place in the history of research and invention.

The published researches of Faraday and of those who followed him in developing our knowledge of electrical phenomena show how greatly they relied upon platinum to provide a means of carrying, making and breaking current. Reliable platinum crucibles played a fundamental part in obtaining analytical data for the infant science of chemistry; platinum boilers permitted the concentration of sulphuric acid in the pioneer days of chemical industry. The development of the electric telegraph, of the incandescent lamp, and later of the thermionic valve, all involved the use of platinum, as did the early internal combustion engines, first for igniter tubes and subsequently for magneto contacts.

Through the development of modern metallurgical knowledge some of these early demands have been met by other materials, but further and broader applications have become established. Today substantial quantities of platinum are employed, for example, as contacts in telephone relays, as electrodes in industrial electrochemical processes, as thermocouples for measuring the temperature of molten steel, and as catalysts in the production of nitric acid and of high octane petroleum spirit. Its resistance to attack and its strength at high temperatures make platinum the only suitable material for certain types of equipment handling molten glass, while platinum-wound electric furnaces are used in metallurgy, in the glass and ceramic industries and in nuclear research.

The association of Johnson Matthey with platinum has been long and intimate. It was in January, 1817, just 140 years ago, that Percival Norton Johnson established himself in business, but five years earlier, at the age of nineteen, he had contributed a paper on platinum to the Philosophical Magazine. By the 1830's the firm had become recognised as refiners of platinum and makers of platinum apparatus, and its steady growth in this field has continued.

A major development in the world's platinum industry began to take shape in the nineteen-twenties, when large deposits of the platinum metals were discovered in the Transvaal in South Africa. Although the problems of extraction presented great difficulties, these were eventually overcome, and in 1931 Rustenburg Platinum Mines Ltd. was formed to work these deposits, with Johnson Matthey as refiners and distributors of the metals produced. To meet the steadily increasing demand steps have been taken from time to time to expand production from these mines, and Rustenburg is now established as the world's largest individual producer of platinum.

Interest in platinum's five sister metals—palladium, rhodium, iridium, osmium and ruthenium—was later in development than with platinum itself, but over the past thirty years or so they too have found growing applications in chemical and electrical engineering.

It is to provide engineers, chemists, metallurgists and other users with a source of current information on the properties and industrial applications of platinum and its associated metals that the publication of Platinum Metals Review has been undertaken.

Platinum Metals Rev., 1957, 1, (1), 2
Platinum Mining in the Transvaal

OPERATIONS AT RUSTENBURG PLATINUM MINES

The mining of platinum in the Transvaal is being carried out by the Rustenburg Platinum Mines Limited, at the two sections of its property, one of which, the Rustenburg section, lies a few miles to the east of the town of Rustenburg while the other, the Union section, is situated some sixty miles to the north of that town.

Rustenburg Platinum Mines Limited has in recent years become the world's largest producer of platinum and is in fact the only major producer whose prime business is the production of platinum group metals.

The deposit worked on both sections of the mine forms part of the so-called Merensky Reef Horizon of that extraordinarily interesting geological formation—the Bushveld igneous complex.

Platinum was first discovered in South Africa in 1923 in lode deposits in the Waterberg district of the Northern Transvaal and the discovery of these occurrences started off a search for further deposits.

The Merensky Reef

Dr. Hans Merensky found platinum in both the Dunite deposits and the Merensky Reef Horizon in the Lydenburg district, and also in this latter horizon in the Potgietersrust area. Both of these discoveries were on the eastern fringe of the Bushveld igneous complex. Dr. Merensky finally located the...
Rustenburg lies some 70 miles to the north-west of Johannesburg in the Western Transvaal. The Rustenburg section of the property lies a few miles to the east of the town, the Union section being some 60 miles to the north.

platinum bearing reef beneath the black turf soil of the Rustenburg district, and this reef has now been traced for over 140 miles in that area.

These discoveries were followed by a minor boom in which several platinum mining ventures were started. The boom was, however, shortlived and following upon reductions in the price of platinum a slump occurred in the late 1920’s. Only two mining companies survived. These companies were Potgieter'srust Platinums Limited and Waterval (Rustenburg) Platinum Mining Company, which in the interests of economy were merged in 1932 to form the Rustenburg Platinum Mines Limited.

In 1947, the Union Platinum Mining Company Limited was formed to work the Merensky Horizon on the Farm Swartklip, some 60 miles to the north of Rustenburg. This company was amalgamated with the Rustenburg company in 1949 and it now constitutes the Union section of Rustenburg Platinum Mines Limited.

Other South African mining houses have prospected, and continue to prospect, areas in the Rustenburg district but no further active mining operations have started.

Geology

The Bushveld igneous complex is a vast composite body of plutonic and volcanic rocks in the central part of the Transvaal. It is irregularly oval in shape, measuring some 280 miles along its major axis with a maximum width of roughly 150 miles, and
covers an area of about 15,000 square miles. The outer or lower portion of the complex consists of norite and allied basic and ultrabasic rocks, and in the inner or upper portion of red granite, granophyre, basalts and pyroclastic volcanic rocks.

The norite forms a great basin-shaped sheet from two to three and a half miles in thickness and the width of the outcrop ranges from five to over twelve miles. The Merensky platinum-bearing horizon is located in the lower portion of this norite sheet.

At both the Rustenburg and Union sections the platinum group metals are mainly concentrated in a layer of coarse felspathic pyroxenitic norite against which lies a three-quarter-inch chromite-rich platiniferous band known locally as the "Chrome Band".

At the Rustenburg section a strike length of approximately 40,000 feet of the reef is being mined with a 9 to 10° dip to the north, whereas at the Union section that portion of the reef being worked has a strike length of about 13,000 feet and an easterly dip of 21°.

On both sections the following minerals are found in the sulphide ore mined:

- Chalcopyrite
- Cubanite
- Nickelliferous pyrite
- Graphite
- Phyrrhotite
- Millerite
- Pentlandite
- Violarite

The primary silicates include olivine, diallage, bronzite and labradorite, while hornblende and biotite occur as secondary reaction minerals.

The metals of economic value recovered...
Four-ton trucks discharging platiferous ore into the underground shaft ore bins

after the reduction and treatment of the ore comprise platinum, palladium, iridium, rhodium, ruthenium, osmium, gold, copper and nickel.

Mining Methods

The dip and strike of the ore bodies on each property are consistent, and although an occasional dyke is met with there is an almost complete absence of faulting. Certain geological disturbances which interfere with mining operations and which exist mainly on the Rustenburg section are circular depressions of the ore body locally known as “potholes”. These vary in diameter from 20 feet to 300 feet and represent a depression of the ore body, usually of about six feet in extent.

The consistency of values found throughout the breadth and length of the ore bodies in each of the two mining areas is remarkable.

In both sections, incline haulages from the surface on or under the reef are used to work the shallower portions of the deposit. These haulages are sited about 2,500 feet apart on strike and are connected by drives on reef. The deeper areas are being opened up by means of vertical shafts and footwall haulages.

Underground traction is by diesel or electric haulage of four-ton hoppers which deliver the broken ore to the shaft ore bins. Very little water is made in the mine and there are therefore no pumping problems. Likewise, ventilation methods are straightforward.

At the Rustenburg section mining has now reached a vertical depth of 1,000 feet from surface, which is equivalent to about 6,000
feet on dip. Three vertical shafts have been sunk to depths varying from 300 feet to 1,500 feet, and a further 1,500 foot shaft is about to be started. The vertical depths of workings at the Union section are of the same order.

Reduction Practice

As the platinum group metals are associated with the sulphides found disseminated throughout the chromite and pyroxenite bands, the collection of these metals follows the collection of the sulphides.

The reduction process may be divided into four sections:

(a) Crushing
(b) Milling and gravity concentration
(c) Concentration by flotation and
(d) Smelting

The smelting process is carried out only at the Rustenburg section and flotation concentrates produced at the Union section are delivered by road to the Rustenburg section.

Two-stage grinding is carried out in ball mills and is followed by gravity concentration on corduroy cloths, the concentrate being cleaned and redressed on James shaking tables ready for transportation as a high grade metallic concentrate to Johnson, Matthey & Co., Limited, of London.

The pulp from this section is thickened and fed to the flotation circuit, the concentrate being itself thickened and filtered on drum filters ready for the smelter section. The moist concentrate is mixed with slaked lime and flue dust and is then briquetted. After drying the briquettes are fed to the blast furnace.
furnaces with coke fuel and fluxes and smelted.

The flow from the blast furnaces is by trap spout to a forehearth where the slag and matte separate. The slag is granulated and dumped, while the matte is tapped at intervals, conveyed to a "Great Falls" type converter and blown to a high grade copper-nickel matte. This matte is cast and either prepared for shipment to the Johnson Matthey refinery in London or sent to Matte Smelters (Proprietary) Limited, which is situated adjacent to the mine at Rustenburg. Matte Smelters (Proprietary) Limited is a joint subsidiary of the Rustenburg Platinum Mines Limited and Johnson, Matthey & Co., Limited.

In the refinery of Matte Smelters the copper-nickel matte is reduced to electrolytic copper and nickel and an enriched concentrate of platinum group metals and gold. The copper and nickel are available for sale either locally or overseas, and the concentrate is sent to London for further treatment by Johnson Matthey.

A pelletising plant, capable of converting moist flotation concentrates into one-inch pellets, is shortly to be installed at the Rustenburg smelter to improve both the handling operations prior to smelting and the smelting process itself.

Labour Force

During the last financial year the two sections together employed about 1,100 Europeans and nearly 9,000 African natives. With the expansion programme in progress these figures will be increased towards the end of 1957 to 1,800 Europeans and 15,000 African natives.

The European labour supply is drawn partly from the farming area around Rustenburg, where the young men are recruited as learner miners, plant operators, etc., while trained miners and artisans are drawn from other mining areas.

The native workers are largely the migratory type who come from areas far afield seeking employment to their liking and in the knowledge that good housing, feeding and medical attention are supplied. Many of these natives complete a number of tours of duty interspaced with long holidays and rest periods at their homes.

Scale of Operations

It has always been the policy of Rustenburg Platinum Mines to operate at a scale commensurate with the demand for platinum, and since the amalgamation of the two sections the company, operating in close conjunction with Johnson Matthey who refine and dispose of the metals produced, has been engaged in a process of almost continual expansion. The annual tonnage milled at the commencement of operations in 1932 was 91,000 tons. By 1955 this figure had risen to 1,600,000 tons and, in view of the present increasing world demand for platinum, a programme is in hand which will result in a milling rate of approximately 2,600,000 tons per annum before the end of 1957.

The present scale of operations involves the consumption of 85,000,000 kilowatts of power and 570,000,000 gallons of water a year, in addition to the purchase of £1,500,000 worth of stores, while £1,100,000 is paid out in European wages and £400,000 to native employees. During the course of the coming year there will be an increase in all these amounts by more than 60 per cent.

The directors of the company are constantly alert to the changing demand for platinum brought about by modern technological development and are ready to enlarge the rate of production when it is required by the developing needs of industry.

(This article has been contributed by the technical advisers to Rustenburg Platinum Mines Ltd., the Johannesburg Consolidated Investment Company Limited.)
**The Protection of Chemical Process Equipment**

**THE USE OF PLATINUM METALS FOR BURSTING DISCS**

By J. M. Pirie, Ph.D., A.M.I.Chem.E.

*It is often impracticable to employ safety-valves for the protection of pressure vessels used for chemical processing. The bursting disc or rupture diaphragm, a dome of metal foil which will burst when the safe working pressure of a vessel is exceeded, is a particularly suitable form of safety device for process equipment. To function effectively, however, the foil disc must possess a combination of chemical and metallurgical properties which for some purposes only the platinum metals can provide. The selection and use of these metals for bursting discs is discussed in this article.*

Any closed vessel which is subjected during use to internal pressure must have some safety device to protect it from damage should the pressure increase beyond control. The oldest and still the most widely used device of this kind is the safety-valve, which has an escape vent normally closed by a conical-seated plug held in place either by a spring or by a dead weight. When the internal pressure exceeds a predetermined level this plug is lifted against the retaining force to open the vent.

The safety-valve is simple and robust but it has a number of serious disadvantages. It is very difficult to prevent leakage between the plug and seat; gummy or resinous fluids can cause the plug to stick in the seat; the working parts cannot easily be protected against corrosive attack; its small relieving area and its high inertia make it ineffective against explosive hazards.

The bursting disc suffers from none of these disadvantages, and at the present time it is becoming widely adopted as a safety device for pressure systems in the chemical industry, and is also being more and more employed in other fields, wherever closed vessels have to be protected against the effect of pressure.

*Fitting a bursting disc assembly into a pipe line*
Standard full-face capsule-type bursting disc assemblies, with pre-domed platinum discs, stainless-steel holders and vacuum supports

It may at first sight seem surprising that such an inherently simple device was not introduced many years ago, and there is little doubt that numerous attempts to develop this form of protection have been made. These attempts were always frustrated by the impossibility of obtaining very thin foil of uniform and reproducible quality, and by a lack of understanding of the behaviour of these foils when subjected to stresses near their breaking point at different temperatures.

The Technical Requirements

To be successful, a bursting disc has to meet two exacting conditions: when the pressure in the system which it protects reaches a predetermined level the disc must burst, and it must withstand without failure the effect of all other lower pressures to which it may be subjected during normal working.

A circular disc of thin ductile foil exposed to uniform fluid pressure on one face assumes a spheroidal form, in accordance with a relationship expressed mathematically by Lake and Inglis (1). The bursting pressure of a disc clamped in an orifice of a given size, held at a fixed temperature, is related both to the thickness of the disc material and to its tensile properties. To obtain discs which will burst at an unvarying pressure it is therefore necessary to make them of an exact and uniform thickness, and to be able to control and reproduce the composition and metallurgical properties of the material.

By skilful workmanship and close control, it is now possible to produce foil from which discs can be manufactured that will with certainty burst within 5 per cent of the chosen pressure, so that the first of the two requirements can be met. The second requirement is governed by more complex factors. Although a disc will at the time of installation withstand at its specified working temperature a pressure up to 5 per cent below the bursting pressure without failure, it is affected during its life by corrosive attack, while the material may creep if the pressure is near the bursting point and may suffer fatigue if fluctuating pressures are applied. All these factors tend to lower the bursting pressure and so to cause discs to fail prematurely in the range of normal working pressures.

Avoiding Premature Failures

Fortunately, premature failure is a nuisance rather than a danger and there are virtually no circumstances in which a disc can withstand without bursting a higher pressure than that for which it is designed. However, repeated premature failures cannot be tolerated if a plant is to operate efficiently and smoothly, and bursting discs could not find general acceptance unless these failures could be prevented.

In process equipment handling corrosive fluids a certain degree of attack can usually be tolerated; for example, in a steel pressure vessel with a designed wall thickness of 1 inch, a corrosion rate of 0.005 inch per year could easily be tolerated if the initial wall thickness were 1 1/2 inch. A bursting disc 0.003 inch thick attacked at the same rate would have
The relationship between bursting pressure and thickness of various metals, in a 2 inch orifice at room temperature.

only half its original bursting pressure after four months in service. There is therefore a need to use materials of exceptionally high resistance to chemical attack and in this respect, since it is quite unaffected by virtually any corrosive condition that would be encountered in chemical process equipment, platinum is pre-eminent. For certain specific chemical conditions, palladium also has the high immunity to attack which is necessary.

At low working temperatures a constant pressure of more than about 85 per cent of the rupturing pressure will cause significant creep in ductile metals. Creep—the plastic flow of a metal under a continually applied force less than that required to cause fracture—is a property strongly affected by temperature. Among the commonly used corrosion-resistant bursting disc metals, such as silver or gold, resistance to creep deteriorates rapidly with increasing temperature and, if they are to be used at much above 100°C, the working load must be restricted to a comparatively small proportion of the bursting pressure, often as little as 50 or 60 per cent, to prevent early failure by progressive thinning. Platinum however has a very high melting point, a high annealing temperature and, similarly, excellent resistance to the effect of creep at temperatures up to about 400°C. Its resistance to the effect of stress-reversals is also adequate for this service.

The comparatively low tensile strength of platinum and palladium is a further advantage in their employment for this purpose. With orifice sizes of say 2 to 6 inches and in the range of pressures encountered in chemical process equipment, the thickness of foil in a bursting disc is necessarily very small, usually in the range 0.001 up to 0.01 inch. To achieve exact thicknesses and to avoid pinholes, the thicker that the material can be, the better. Because of its higher
tensile strength, a disc of a base metal such as nickel is only about half as thick as one with the same bursting pressure made of platinum. The advantages of platinum for bursting discs are in fact so evident that one of the earliest papers dealing with bursting disc materials, by M. E. Bonyun (2), gives details of tests from which platinum was found to be the most suitable metal available. Its successful use has, however, entailed considerable development work, because, although both platinum and palladium can be prepared in a state of very high purity and foil can be rolled under close metallurgical control, they have one disadvantage in that the pure metals tend to have a coarse grain structure and, when taking a spheroidal form under the application of fluid pressure, they tend to develop a granular “orange peel” surface which gives rise to inconsistent bursting pressures.

Although pure metals are in general preferred to alloys as bursting disc materials because of their greater susceptibility to close control, very accurate addition of minor constituents to the platinum metals is pos-
sible, and a series of alloys has been developed containing small additions of other noble metals. These alloys have a fine grain structure and can be rolled down to very thin foil that bursts at accurately reproducible pressures. The relationships between the bursting pressure and thickness of two platinum alloys and one palladium alloy, and also of aluminium, silver and nickel, are shown in the graph on page 11.

**Disc Assemblies**

A bursting disc cannot be considered alone; it is essentially part of a complete assembly and for accurate performance it must be fitted into a correctly designed holder. It is the general practice to pre-dome the disc by applying a hydraulic pressure of up to 90 per cent of the required bursting pressure. When the bursting pressure is subsequently applied, the disc stretches still further and eventually ruptures by tensile failure at the thinnest part of the crown. The ruptured disc opens petal-fashion, all the metal being retained at the periphery, so that it can all be recovered and the value reclaimed. The pre-doming process is an effective aid to inspection since it clearly reveals any surface imperfection or incipient pinhole in the disc.

It is rarely necessary to make the whole assembly of a noble metal, although silver-sheathed holders are often used in service with chlorine. It is generally possible to select a base metal, such as nickel, Monel metal or stainless steel which is sufficiently resistant for the construction of the holder and the vacuum support. This support is necessary when a disc assembly is exposed to both positive and negative pressures, since without it the domed disc would be inverted under negative pressure and with only a few reversals would be sufficiently weakened to burst. Two of the accompanying figures show assemblies with platinum discs mounted in stainless steel holders and with stainless vacuum supports.

These flanged holders are used for orifice sizes from 1 inch upward. For the many smaller assemblies used to protect laboratory autoclaves and the like, in which platinum discs are normally employed, the cone-ring holder shown in the accompanying figure is most suitable.

If large discs to burst at high pressure are required, a disc solely of platinum might require a considerable investment, but it is practicable to manufacture composite discs in which the face in contact with the working fluid is a thin platinum foil, backed up by a thicker disc of another metal such as nickel that bears most of the stress. The bursting pressure of such a composite disc is the direct sum of those of the individual parts.

**Applications**

In chemical plant services, platinum has been principally employed in contact with hydrochloric acid vapours and chlorinated hydrocarbons, and under less corrosive conditions where the prevailing temperature is high. Patrick (3) describes a system in which platinum discs are used to protect a system containing superheated water. In certain chemical works where numbers of disc assemblies are installed the practice has been adopted of using platinum or palladium for general service, even under mildly corrosive conditions, with the purpose of having a single disc material for all services. This is preferable to using a variety of materials and by so doing entailing the risk of premature failure or accident through selection of the wrong material for a particular purpose.

**References**

2 M. E. Bonyun ... ... ... *Trans. Amer. Inst. Chem. Eng.*, 1935, 31, pp. 256-272
Electrodeposited Rhodium in Co-axial Radio-Frequency Circuits

By D. O. Walter, A.M.I.E.E.

Rhodium is widely used over a silver electrodeposit on the current-carrying and sliding contact surfaces of co-axial line assemblies and also as a general protective treatment. In this article recommendations for thickness of deposits are given, together with some suggestions on design of the contact elements to achieve long life and consistent performance.

The extensive use of radio as a navigational aid has resulted in the employment of higher and higher frequencies or, in the more usual phrase, of shorter wave lengths. At these high frequencies, corresponding with only centimetric wave lengths, it is no longer possible to employ normal types of circuits consisting of coils and capacitors, and use must be made of co-axial conductors or lines, generally fabricated from rigid tubular members. These co-axial circuits are displaced by wave guide elements only at still higher frequencies.

In co-axial circuits the electrical parameters are established by the mechanical dimensions and by the electrical conductivity of the current-carrying surfaces. The dimensions of the circuit—either the internal volume of a cavity or the actual electrical length of a co-axial line—frequently have to be adjustable, and this adjustment is usually achieved either by having a section of the line made telescopic in form, or by arranging movable contact elements which short the inner member of the co-axial line to the outer.

While such mechanical assemblies offer few problems in the experimental or laboratory stages, they can present major design problems in apparatus subjected to continuous use and to atmospheric conditions which can frequently be corrosive. Successful operation is critically dependent on the stability of electrical contact performance and on freedom from fluctuations of contact resistance during the adjustment of the apparatus.

It is well known that at high frequencies substantially all the current flowing in a circuit is confined to the surfaces of the conductors. In the case of a co-axial line, therefore, practically all the current flows on the outside of the inner conductor and on the inside of the outer one. In resonant circuits having a high Q value the magnitude of the current can, of course, be high, and the losses in the circuit can also be high if the current-carrying surfaces are insufficiently good conductors. The Q value of any resonant coil or line is inversely proportional to the circuit resistance and it follows that for the highest value of Q the total circuit losses must be reduced to a minimum. By far the greatest loss in a well-designed resonant line is resistive, and it becomes necessary to employ high conductivity current-carrying surfaces.

The obvious choice for these surfaces is silver, and the most usual procedure is to electrodeposit the silver on to a suitable base metal assembly. Care must be taken, however, to ensure that the deposit is hard, of fine grain, and of a type giving a low radio-
frequency resistance. The thickness of the silver electrodeposit is in practice not unduly critical. A thickness of 0.0005 inch (12.5 μ) is generally adequate for radio-frequency purposes, although thicker deposits are often used as a means of obtaining added protection in corrosive atmospheres.

Silver having been chosen as a surface conductor on purely electro-technical grounds, it now becomes necessary to ensure its suitability in practice, and two difficulties emerge. First, silver, while not subject to oxidation, is readily tarnished by sulphur compounds and is attacked by salt spray or sea water immersion. Thus it becomes necessary to give the silver a protective coating which will not adversely affect the radio-frequency performance.

Secondly, while silver-to-silver forms an excellent contact combination from an electrical point of view, the rubbing or sliding of two silver surfaces can lead to galling and rapid self-destruction of the surfaces. Indeed, the mutual sliding of two clean silver surfaces can lead to partial cold welding. Here again, there is need of a further surface treatment to overcome the mechanical difficulties and to provide two contact surfaces which remain electrically clean and undamaged throughout their life.

A very thin electrodeposit of rhodium on to the silver surfaces can meet all these requirements, provided that certain factors are borne in mind when designing the apparatus.

**Freedom from Oxidation and Tarnish**

Rhodium, one of the six metals of the platinum group, is a very hard, white metal, entirely free from oxidation or tarnish, and possessing remarkable resistance to wear. Certain of its physical properties are given in the table below. Its complete freedom from film formation makes rhodium an excellent contact material where there is no appreciable voltage, and provides a very stable, as well as a low, contact resistance. Some indication of the order of contact

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**Properties of Rhodium**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific gravity</td>
<td>12.4</td>
</tr>
<tr>
<td>Melting point, °C</td>
<td>1960</td>
</tr>
<tr>
<td>Thermal conductivity, C.G.S. units</td>
<td>0.36</td>
</tr>
<tr>
<td>Resistivity, microhms per cm cube at 20°C</td>
<td>4.7</td>
</tr>
<tr>
<td>Temperature coefficient of resistance (0 to 100°C)</td>
<td>0.0046</td>
</tr>
<tr>
<td>per °C</td>
<td></td>
</tr>
<tr>
<td>Thermal e.m.f. against platinum, (0 to 100°C) microvolt</td>
<td>7.0</td>
</tr>
<tr>
<td>per °C</td>
<td></td>
</tr>
<tr>
<td>Vickers hardness (electrodeposited)</td>
<td>800</td>
</tr>
</tbody>
</table>

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*Platinum Metals Rev.*, 1957, 1, (1)
resistances obtained with electrodeposited rhodium is given in Fig. 1, which shows a series of determinations, employing point contact, in which variations in contact pressure from 1 to 400 grams produced a change in contact resistance of only 5.0 to 1.4 milliohms.

For purposes of clarifying the two problems, that of protection and of contact performance, they will be treated separately although they are, of course, intimately related.

Protection of Silver Surfaces

The simple protection of silver surfaces against tarnish and discoloration due to handling is readily achieved by depositing a very thin film of rhodium, and practice has shown that for extended exposure to industrial atmospheres a rhodium thickness of 0.000015 inch (0.375 μ) should be regarded as a minimum, although 0.00002 inch (0.5 μ) is more usually employed. If, however, the components are liable to be subjected to much heavier attack, such as for instance by salt spray, somewhat heavier rhodium deposits are necessary, and in extreme cases heavier silver underlays are desirable.

The electrodeposition of rhodium should follow as soon as possible after the deposition of the silver undercoat, and it is usual to silver and rhodium plate the whole of a radio-frequency component in addition to the current-carrying surfaces.

Provision of Sliding Contact Surfaces

Under certain conditions, electrodeposited rhodium can give exceptionally satisfactory electrical performance for long periods of use, but, as stated previously, for the best results certain factors must be considered in the design of the circuit components.

It must be realised that electrodeposited rhodium has a hardness of around 800 VPN, and that the deposit usually has little thickness. In view of this great hardness compared with any of the usual base metals, and in particular with the silver undercoat (which normally has a hardness of 100 VPN), care must be taken to avoid high point-loading of the rhodium film. Excessive loading of this sort will fracture the rhodium deposit, exposing sharp cutting edges which will rapidly destroy both rhodium and silver deposits. (The effect may be admirably demonstrated by pressing a marble into the sugar icing of a cake). Fortunately this situation can be avoided by maintaining the radius of contact zones sufficiently large, by applying contact pressures by means of low-rate spring elements, and by reducing contact pressures.

The last statement would appear to be contrary to normal practice where good contact performance is demanded, but it must be remembered that rhodium is completely free from tarnish or other films, and that the contact resistance between two rhodium plated surfaces is reaching its stable value with pressures of 50 to 100 grams (Fig. 1). A typical resistance value at the latter pressure would be approximately...
0.002 ohm. Bearing in mind that for sliding contact surfaces the rhodium deposit is heavier than previously specified for protection, the pressures indicated above can safely be used without incurring damage.

For a multi-fingered sliding "short" for a co-axial line the desirable thickness of rhodium will, of course, be dependent on the anticipated life and frequency of sliding. For the surface of the lines deposits of 0.0002 inch (5 μ) to 0.0005 inch (12.5 μ) thickness are normal, while the tips of the contact fingers, which get the greatest wear, may have a deposit as thick as 0.001 inch (25 μ). Deposits in excess of 0.001 inch are seldom used owing to the difficulty of maintaining a bright smooth surface.

As a measure of economy it is customary to increase the thickness of the rhodium only over those areas where sliding contact takes place, the remainder of the surfaces being given the protective treatment already discussed.

Design Considerations

To attempt to deal with the design considerations for all types of co-axial line components would be far too complex and lengthy a matter, and it is proposed to consider by way of example a typical shorting assembly as manufactured by two alternative methods. The various design points dealt with will be applicable to other types of components, and should form a sound basis for sliding contact design.

In that class of apparatus where stability and performance are paramount, ease of manufacture and cost being of secondary importance, the sliding shorts or "top hats" are usually machined from the solid or fabricated from tubular elements in phosphor-bronze or beryllium-copper. The general appearance of these parts is as shown in Fig. 2, in which a section view of one half of a shorting bridge is given.

It will be noted that the walls of the components have been thinned down over substantially their full length so that each finger of the contact has adequate flexibility, independent of the neighbouring fingers, to give the desired contact pressure, and for this pressure to be only slightly modified by variation of finger deflection. In other words the fingers have a long-law characteristic.

(The appendix gives the well known formulae for calculating deflection and stress in flat cantilever springs, which are sufficiently close approximations to the separate finger springs under discussion.)

The diameter of the top hat is greatest at the contact end by such an amount that insertion into the outer tubular member of the line will produce the desired individual contact finger deflection. The base end of the assembly is somewhat less in diameter than the bore of the outer tube. Under these conditions slight variation of tube diameter or slight irregularity of shape or alignment of the top hat operating mechanism will produce substantially no variation in contact pressure.

Having established the conditions of contact pressure, it is now necessary to consider the shape of the contact surfaces. There are three design factors to be considered in this connection. First, where two
rhodium plated surfaces are in rubbing contact it is always desirable for one of these contacts, in this case the finger contacts, to be dome shaped, but the radius of doming must be large. A small dome will lead to rapid wear owing to scoring of the surfaces by the cutting edge formed as the contact zone wears. Typical minimum radii are usually of the order of 0.125 inch (3 to 4 mm).

Secondly, reference to Fig. 3 will show the necessity of shaping the contact areas of the springs in both axial and circumferential senses where these springs bear on the inside of a tubular line. Insufficient care in achieving such a form will lead to very rapid scoring and to failure of the contact surfaces. In the case of contacts bearing on the outer surfaces of lines there is, of course, no problem, as a cylindrical contact surface lies with its axis tangential to the tubular member.

It might seem that the various simple design factors mentioned above are unnecessarily elaborate and represent a penalty to be paid when using rhodium contact surfaces. This is not so, as all the points mentioned are valid whatever contact material is used, and only represent good design. It is true, however, that failure to observe these points will nullify the very considerable advantages offered by rhodium contact surfaces in the type of apparatus under discussion.

Thirdly, it is obviously desirable in precision apparatus to have the point of contact to the co-axial line as near as possible to the tips of the contact fingers, for it is here that the current path suffers a discontinuity in passing from the co-axial surface to the inner surface of the top hat. It will be seen in Fig. 2 that the lengths of the top hat fingers are made to approximate to $\lambda/4$, or of course to $N\lambda/4$ where $N$ is odd, as at these points in a resonant line the current flowing is a minimum. Frequently it is not possible to achieve a full quarter-wave in the top hat, but there is real advantage in approaching as near the quarter-wave length as possible.

The choice of the number of fingers to

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**Design Formulae**

The well-known formulae for deflection and stress in simple flat springs of rectangular cross-section, clamped at one end, are as follows:

$$\delta = \frac{4 P l^3}{E b t^3} \quad \text{and} \quad f = \frac{6 P l}{b t^3}$$

where $\delta$ = deflection in inches $\quad b$ = width in inches

$P$ = load in pounds $\quad t$ = thickness in inches

$l$ = length in inches $\quad f$ = working stress in pounds per sq. in., normally approximately 75 per cent of proof stress (0.1 per cent extension)

$E$ = Young’s modulus in pounds per sq. in.

The following values for Young’s modulus and for proof stress may be considered typical:

- Beryllium-copper (heat treated) $E = 18 \times 10^6$ pounds per sq. in.
- Proof stress $= 150 \times 10^3$ pounds per sq. in.
- Phosphor bronze (BS 407/2—Hard) Proof stress $= 62 \times 10^3$ pounds per sq. in.
be used will, of course, depend on the dimensions of the lines in question. For very small diameter lines only a few contact fingers are possible by virtue of the small physical dimensions. It is, however, always desirable to keep the number of contacts large so as to obtain stability of contact performance, and more important still to ensure, as nearly as possible, continuity of the skin current sheet and freedom from excitation of the back end of the line.

As a guide, a line of 1 inch diameter (25 mm) will usually have some 30 fingers, correspondingly more being used for larger diameters.

**Quantity Production**

In the less critical type of apparatus, and where quantities demand a simplified manufacturing technique, it is now common practice to fabricate sliding shorts from strips of spring material, pre-slotted, formed, electroplated, polished, and finally wrapped round and soldered to a solid back plate.

The general principle is self-explanatory and is illustrated in Fig. 4. The fact that the diameter on which the spring strip is wrapped may be such that there is not an integral number of fingers round the circumference is of little importance, provided that the fingers are small in width, as a gap between the ends of the strip of half-a-finger width has little or no effect on performance.

The advantages of this method of construction are obvious, as the forming, slotting, plating and polishing processes are carried out while the material is in the flat state, thus simplifying handling problems.

It is customary to silver plate the strip all over its surface, but to restrict rhodium plating to the spring fingers only, and to restrict the heavy rhodium to the spring finger contact areas.

**Electrodeposition of Ruthenium**

Very little work has been carried out on the electrodeposition of ruthenium although it is known that deposits have been obtained from dilute solutions of ruthenium nitroso salts. An investigation recently completed at the Atomic Energy Research Establishment, Harwell, by A. C. Littlejohn (A.E.R.E. C/R 1892, 1956) shows that, under certain conditions, uniform deposits of ruthenium may be obtained on copper cathodes from solutions of ruthenium nitroso trichloride in dilute hydrochloric acid.

Numerous runs were carried out, using a platinum anode, at constant potential, but it was found in all cases that once an initial coating of ruthenium had been deposited at low current density, the current gradually increased to very high values and brittle deposits were obtained. Constant potential electrolysis was therefore dropped in favour of constant current electrolysis. In these experiments current densities up to 20 mA/cm² were used, and the deposits were considerably improved.

Conditions finally recommended for successful deposition include a solution of $5 \times 10^{-3} \text{ M } \text{Ru(NO)Cl}_3 + 0.5 \text{ N HCl}$, and a current density between 2 and 5 mA/cm².
A Thermocouple for High Temperatures

ADVANTAGES OF THE 'FIVE-TWENTY' COUPLE

By J. C. Chaston, Ph.D., A.R.S.M.

For a great many years the thermocouple formed by connecting pure platinum to the alloy of platinum with 13 per cent of rhodium has been the most widely used of all noble metal couples. The reliability of this couple is such that it has been adopted as the standard by which temperatures between 660°C and the melting point of gold (1063°C) are established on the International Temperature Scale, and it is of course widely used throughout industry, particularly in determining the temperature of molten steel.

If need be, and if sufficient precautions are taken, this couple can be used to measure temperatures accurately up to just below the melting point of platinum, 1769°C, and indeed it can be calibrated at the melting point of platinum by heating carefully in a gradient furnace until the platinum wire of the couple melts back at the junction.

However, at temperatures over about 1650°C the platinum wire becomes weak mechanically and needs to be carefully supported to avoid creep failure under quite low loads, so that it is not generally practicable to use the couple—even for single-immersions in liquid steel—at temperatures much above 1650°C, and for regular temperature recording or control it is usual conservatively to regard 1500°C or 1550°C as the safe upper limit.

The majority of industrial high temperature operations fall within the range covered by this standard couple, but there is, of course, a small demand—and one which is likely to grow rather than to diminish—for a thermocouple capable of working consistently and satisfactorily at higher temperatures. A great number of thermocouple combinations has been proposed for such applications, but none is entirely satisfactory. Some, such as the tungsten-molybdenum and the tungsten-iridium thermocouples, need to be protected from oxidation and nearly all of them—including the frequently mentioned Feussner couple between iridium and a 40 per cent iridium-rhodium alloy—are either very brittle and difficult to install or tend to embrittle.

Fig. 1—Liquidus-solidus curve for rhodium-platinum alloys
seriously in use. Moreover, many are unstable in service, generate only a very minute e.m.f., or develop inflected e.m.f.-temperature curves.

For these reasons, attention has recently been re-directed to the possibility of using two rhodium-platinum alloys as a thermocouple element. The addition of rhodium to platinum is known to raise the solidus temperature, and although the shape of the solidus curve is not well established, it is generally thought to be somewhat as shown in Fig. 1. Thus, by substituting a rhodium-platinum alloy for pure platinum, it is obviously possible to increase the range of temperatures that can be measured before one of the wires starts to melt.

The e.m.f. developed between two rhodium-platinum alloy wires is, of course, less than that between the alloy richest in rhodium and pure platinum. It is, in fact, equal to the difference between the e.m.fs. developed by the two couples, one between the richer alloy and pure platinum and the other between the poorer alloy and pure platinum at any temperature. The e.m.f. developed by any combination at 1000° C can thus readily be derived from Fig. 2.

The problem which thus remains is to choose a couple combination such that:

1. The melting point of the wire poorer in rhodium should be as high as possible.
2. The e.m.f. developed at the working temperature should be as high as possible.

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*Fig. 2—E.m.f. of rhodium-platinum alloys against platinum at 1000° C*

*The 5:20 thermocouple is used in testing refractory bricks under compressive load at high temperatures in the Dorman Long research department*
The two wires should be ductile and easy to fabricate and use.

The first condition requires that all wires should be as rich in rhodium as possible, but the third condition limits the composition of the wire richest in rhodium to the alloy containing about 20 per cent.

In order to obtain as high an e.m.f. as possible it seems desirable that the wire poorer in rhodium should not contain much more than 5 per cent of rhodium, although rather more could be tolerated with sensitive measuring instruments. On the whole, however, it would appear that the 5 per cent rhodium : 20 per cent rhodium couple is about the most desirable combination obtainable, and experience with this couple in practice has confirmed its value as a high temperature thermocouple. It can be used continuously at temperatures up to about 1700°C, while its absolute limit for single determinations is set by the melting point of the 5 per cent alloy, which is approximately 1825°C.

The above reasoning, however, is not quite the whole story. The use of 5 per cent rhodium-platinum alloy in place of platinum brings with it three added advantages which are not always appreciated. One is that the alloy addition very greatly increases the strength of the platinum wire at high temperatures and almost entirely eliminates the danger of fracture by creep at temperatures above about 1700°C. Moreover, the couple consisting of two rhodium-platinum alloys is more tolerant of small amounts of contaminating elements in service. It has the added advantage that the room temperature e.m.f. is negligible so that for many purposes a controlled cold junction and special compensating leads need not be used.

All in all, therefore, the 5 per cent rhodium-platinum : 20 per cent rhodium-platinum thermocouple represents a notable advance in the provision of thermocouples suitable for measuring very high temperatures. It has found an increasing use in measuring the temperature of open-hearth furnace roofs, for example, and in the manufacture and testing of refractories. The illustration on page 21 shows a test-rig in the research department of Dorman Long and Co. Ltd. for investigating the behaviour of refractory bricks under compressive load. Formerly this work was carried out with an optical pyrometer, but the use of the 5 : 20 couple has given greater precision in the range 1600 to 1730°C in which failure occurs.

This new tunnel kiln, firing basic refractory bricks at temperatures up to 1650°C at the Worksop plant of General Refractories Ltd., is equipped with 5 : 20 rhodium-platinum thermocouples.
Creep Properties of Platinum Metals and Alloys

A number of industrial applications of the platinum metals involve their subjection to long-term stresses at high temperatures, and for the engineer responsible for design considerations in these fields there is a need for more data on the creep properties of these metals and their alloys. The use of platinum alloy catalyst gauzes up to three metres in diameter but constructed of wire only 0.04 to 0.06 mm. in diameter, operating at around 800°C, of platinum crucibles holding several litres of molten optical glass at about 1400°C, and of platinum bushings for the manufacture of glass fibre at temperatures up to 1500°C, are examples of such uses.

A survey of available data on this subject, together with some original work, has recently been presented by Dr. G. Reinacher of Degussa, Hanau (Metall, 1956, 10, (13/14) 597–607). This gives first the results of room-temperature creep tests on palladium, platinum and 10 per cent rhodium-platinum in the annealed condition, while in the latter part of his paper the author reviews the relatively sparse literature on the creep properties of the platinum metals at high temperatures and evaluates them on a common basis.

Room Temperature Tests

The conventional proof stress (0.2 per cent extension) was first determined, and then the standard German 45-hour short-time creep limit, the stress necessary to produce a permanent extension of 0.2 per cent after 45 hours. Similar tests extending to 1,000 hours were carried out, and the three sets of values were plotted against time and extrapolated to 10,000 hours. The results are summarised in the table, from which it will be seen that, when in the annealed condition, the three materials tested behave similarly to some materials of much lower melting point in that they creep at stresses well below the 0.2 per cent proof stress. The interesting point emerges that while the proof stress does not itself form a sound basis for design, the ratio of 10,000 hour creep stress to proof stress remains reasonably steady and could provide a means of arriving at a safe loading.

High Temperature Data

In the remainder of the paper, published short-time and long-time creep data are reviewed in an endeavour to establish worthwhile practical information. As a result, some useful data—which should, however, be treated with caution if applied in practice—have been derived for the creep properties of platinum up to 1300°C, of palladium, rhodium and iridium at 1000°C, and of platinum and its binary alloys with 10 per cent rhodium and iridium at 1100°C.
The Platinum Metals in Catalysis

PAPERS PRESENTED AT INTERNATIONAL CONGRESS

A wide range of reactions, studied from both theoretical and practical points of view, was discussed at the International Congress on Catalysis held in Philadelphia in September. Extending over five days, and attracting some eighty papers, the Conference was organised by the Catalysis Clubs of Philadelphia and Chicago, the National Science Foundation, the University of Pennsylvania and the International Union of Pure and Applied Chemistry. The platinum metals were employed as catalysts in many of the investigations described, and significant results were reported on the lesser-known catalytic properties of rhodium and ruthenium.

Smith and Thompson (1) found, for instance, that rhodium had an important advantage over platinum as a catalyst in the hydrogenation of methoxybenzenes. Anisole, the three dimethoxybenzenes and \( \text{I, 2, 3} \), trimethoxybenzene were hydrogenated over both Adams' platinum catalyst and 5 per cent rhodium on alumina, and it was found that rhodium could hydrogenate the aromatic ring with far less cleavage of the methoxy groups (6-18 per cent) than that caused by platinum (40-60 per cent). The amount of cleavage was found to increase linearly with temperature for each catalyst, except in the case of anisole where the change with temperature was quite small or negligible. The discovery that rhodium, particularly at lower temperatures, can catalyse the reduction of the aromatic nucleus with only slight cleavage of the methoxy groups should be of importance in organic syntheses. The rate constants for the hydrogenation with pure platinum oxide were only two to three times greater than with 5 per cent rhodium catalysts, indicating that rhodium must have a greater activity per unit weight than platinum.

Hydrogenation was therefore attempted with pure rhodium oxide, but no reaction occurred, due probably to the conditions being insufficient to reduce the oxide to the metal form.

The use of rhodium and ruthenium as catalysts in liquid-phase hydrogenation by Gilman and Cohn (2) emphasises their superiority over palladium and platinum for certain specific reactions. Carrier-based rhodium catalysts should find a large scale application in the reduction of substituted aromatic ring systems; the hydrogenation of rings containing alkyl-, hydroxyl- or carboxyl substituents was stoichiometric, and in no case was any evidence of cleavage found. Substituent groups on a heterocyclic nucleus are similarly immune from cleavage in rhodium catalysed hydrogenation, and unsubstituted heterocyclic compounds such as pyrrole and pyridine, which had resisted previous attempts at catalytic hydrogenation over nickel, were easily reduced in the presence of rhodium catalysts.

Ruthenium in Hydrogenation

A ruthenium catalyst consisting of 5 per cent metal on either activated alumina or charcoal was found to be specific for the hydrogenation of the carbonyl group in aliphatic aldehydes and ketones under atmospheric conditions. With this catalyst preferential reduction of the carbonyl group in the presence of an olefinic linkage occurs, so that in certain instances unsaturated alcohols can be obtained. Ruthenium is also especially effective in the conversion of sugars to polyhydroxy alcohols. At elevated temperatures and pressures the disaccharides, sucrose and lactose, are both hydrolysed and reduced giving dulcitol, sorbitol and mannitol. Maltose
is not hydrolysed and is only reduced with difficulty using large amounts of catalyst, the product being maltitol. The hydrogenation of dextrose was studied in detail and the reaction rate was found to increase sharply with pressure. These investigations show ruthenium to be of particular interest as a catalyst for the reduction of saccharides.

**Oxidation Catalyst Films**

An interesting paper by Adey and Calvert (3) describes the preparation of an oxidation catalyst coating suitable for deposition on non-porous surfaces such as metals. This has been commercially applied in the coating of resistance wire, which is then wound into an electrically energised coil giving the catalyst in a convenient form for bringing to activation temperature. This film is more active as an oxidising catalyst than a straight deposit of platinum, has a longer life and is less subject to poisoning. It has already found commercial use as a unit for deodorising the steam and grease from electric ovens; it is placed in the oven-vent and oxidises the gases as they pass over it.

Evaporated metallic films were used as catalysts by Anderson and Kemball (4) in their work on the catalytic exchange and deuteration of benzene. They used films of palladium and platinum (among others), the most extensive results being obtained with these two metals because it was found that on these surfaces the exchange and deuteration reactions occurred simultaneously.

**Influence of Atomic Character**

A contribution to the problem of the nature and extent of a relationship between catalytic activity and position in the periodic table (character of the metal bond) was submitted by Amano and Parravano (5) who used alumina-supported ruthenium, rhodium, platinum and palladium as catalysts in the vapour-phase hydrogenation of benzene. Catalytic activity was found to be in the order Rh > Ru > Pt > Pd; with the exception of palladium, this sequence is in accord with the behaviour previously established for compact metals and films.

**Reforming Catalysts**

In a paper dealing with the basic activity properties of reforming catalysts Weisz and Prater (6) attempted to correlate the two basic functional activities of dual-function catalysts to their performance in naphtha reforming. Catalyst samples of different platinium activity were prepared, and also

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**References**

1. H. A. Smith and R. G. Thompson

2. G. Gilman and G. Cohn
   - The Action of Rhodium and Ruthenium as Catalysts for Liquid-Phase Hydrogenation.

3. W. M. Adey and W. R. Calvert
   - The Preparation and Use of an Oxidation Catalyst Film for Non-Porous Supports.

4. J. R. Anderson and C. Kemball
   - Catalytic Exchange and Deuteration of Benzene over Evaporated Metallic Films in a Static System.

5. A. Amano and G. Parravano
   - The Vapour-Phase Hydrogenation of Benzene on Ruthenium, Rhodium, Palladium and Platinum Catalysts.

   - Basic Activity Properties for Pt-type Reforming Catalysts.

7. G. C. Bond and J. Addy
   - The Reaction of Cyclopropane and of Propane with Deuterium over Metals of Group VIII.

those in which the activity of the support was varied by altering its composition. Catalytic reaction tests were devised for measuring each of these activities separately, and the results indicate that above a certain critical platinum activity the reforming power of the catalyst depends on the "acidic" activity provided by the promoted support.

Pumice-supported palladium, rhodium, platinum and iridium were used by Bond and Addy (7) in an investigation into the reaction of cyclopropane and propane with deuterium. The order of activity for both reactions was found to be Rh > Pt > Pd; however this may have no basic significance since it was measured per unit weight of metal instead of per unit surface area. Preliminary results were obtained for iridium and its behaviour was expected to be closely similar to that of rhodium.

Cathodic Protection of Naval Vessels

In a paper on "Navy Experimental Work with Cathodic Protection" presented to the National Association of Corrosion Engineers (Corrosion, 1956, 12, 18-24), Irving D. Gessow describes the work done by the United States Navy Bureau of Ships on the cathodic protection of ships' hulls. Impressed current systems using graphite rod anodes are satisfactory for the external protection of inactive ships, and may increase the docking interval from five to seven or eight years.

No clear evidence is yet available as to the economic advantage of the general use of cathodic protection for active ships, but work has been concentrated principally on destroyers and submarines, where the pitting of plates may have serious consequences. Both sacrificial and impressed current systems have been fitted; magnesium anode sacrificial systems are relatively heavy and require replacement in two years or less while impressed current systems with either graphite or platinum-clad anodes are lighter and more permanent but are higher in first cost. One destroyer and one submarine have been fitted with platinum-clad anodes. The submarine installation appears very successful; results from the destroyer installation were not available at the time the paper was prepared, but it is understood that an inspection of the destroyer after fourteen months of service showed that the anodes were clean and bright and apparently performing satisfactorily, while in general the hull was in excellent condition.

One of seven platinum-clad anodes installed on the hull of a destroyer. Each anode has a platinum sheath 0.005 inch thick on a silver alloy rod 50 inches long.
ABSTRACTS

of current literature on the platinum metals and their alloys

PROPERTIES

Precipitation Hardening in Platinum-Iridium Alloys
E. RAUB and W. PLATE, Z. Metallkunde, 1956, 47, Oct., 688-693

Describes X-ray and microscopic investigations on the whole range of Pt-Ir alloys after differing heat treatment together with resistance and expansion measurements on the alloys containing 10-50% Ir. The Pt-Ir alloys are shown to solidify without exception in solid solution with a broad miscibility gap at lower temperatures having its highest point at about 975° C and 50% Ir and extending from 7-99% Ir at 700° C. Precipitation hardening results from this immiscibility in alloys containing 10-90% Ir; however, as equilibrium is reached only very slowly, this is difficult to detect by X-rays and becomes ultimately impossible below 700° C. Hardness and resistivity changes are observed after short tempering periods.

The System Uranium-Palladium

The experimental procedures used in investigating the system U-Pd are described. The results are illustrated by a phase-diagram and photomicrographs and show the presence of a eutectic between U and U Pd, a stable compound UPd3 and two other compounds UPd and U5Pd6 formed by peritectic reaction. The solubility of Pd in U is slight, but up to 38% U dissolves in the δ solid solution which forms from UPd3 by peritectic reaction.

The Electronic Properties of Ni-Pd Alloys

A theoretical discussion of the magnetic properties of Ni-Pd alloys based on the collective electron treatment of ferromagnetism together with suggestions for suitable experimental work to test the theoretical conclusions.

Preliminary Electrical-Resistivity Measurements of the Ni-Pd Alloy System

Describes electrical resistivity measurements on a range of Ni-Pd alloys at various temperatures. The results show a maximum resistivity at about 70%, Pd and the theoretical implications of this are discussed.

The Hall Effect in the Ag-Pd Alloy System

Room temperature Hall coefficient measurements on the Ag-Pd alloy system are described; divergences from normal behaviour are obtained which cannot be explained using any of the known multiband models for the Hall coefficient.

Decay of Neutron-Deficient Isotopes of Pd and Rh
S. KATCOFF and H. ABRASH, Phys. Rev., 1956, 103, No. 4, 966-970

Pd isotopes of masses 98, 99 and 101 were prepared by α-particle bombardment of Ru. The radiations of these isotopes and of their Rh daughters—masses 98, 99 and 101—were investigated with a scintillation coincidence spectrometer.

ELECTROCHEMISTRY

Investigation into the phase-group impedance in noble metal electrodes in the region of hydrogen adsorption

III “New” activated Rh and Au electrodes

Experiments performed on these new electrodes are described. The new Rh electrode is shown to behave differently from Pt, Ir and the older type of Rh electrode in that it has a lower Hz adsorption, and the graphs relating Hz overvoltage to resistivity etc. also show significant differences.

The Mechanism of the Electrolytic Evolution of Oxygen on Pt
J. O’M. BOCKRIS and A. K. M. SHAMSHUL HUQ

Describes work on the kinetics of the electrolytic oxygen evolution reaction on bright Pt anodes in H2SO4 solutions under ultra-pure conditions over the concentration range 10-3 to 10-1 N and in the presence of varying amounts of Na2SO4. The current density ranged from 10-13 to 10-3 A cm-2. Investigations were also carried out on the
attainability of a reversible oxygen electrode, the time variation of oxygen overpotential at constant current density, its decay with time on open circuit and the cathodic ionisation.

ELECTRODEPOSITION

Some Experimental and Practical Aspects of Heavy Rhodium Plating

Describes the results of laboratory work carried out on the thick plating of Rh from sulphate and phosphate-sulphate electrolytes, together with some exploratory work on phosphate, fluoroborate and sulphamate baths. Experimental techniques are described for the preparation of the electrolytes and the examination of the deposits with reference to thickness, adhesion to basis metal, surface condition, incidence of cracking, internal stress and hardness.

Details are given of the results obtained for the sulphate and phosphate-sulphate electrolytes in terms of quality of the deposits under varying deposition conditions. The effect of variants in the basic methods of preparing the electrolytes was studied and it was found that, except in the case of hardness, there was no correlation between the properties of the deposit and the various methods of preparation examined. In both electrolytes the freshly prepared solutions gave very highly-stressed deposits showing a high incidence of cracking and had low cathodic efficiencies, but after ageing the cathodic efficiencies improved together with the quality of the deposits. These results showed that for thick Rh plating the acid sulphate electrolyte has advantages over the phosphate bath in respect of a higher cathodic efficiency together with rather lower internal stress and greater hardness of the deposits. It is also easier to maintain since the quality of the deposit is very little affected by the build up of free sulphuric acid which occurs when the bath is replenished. Of the other three electrolytes studied the exploratory results indicate that the phosphate and fluoroborate baths have no advantages over the sulphate, but that the sulphamate bath merits further consideration since it offers a wider range of deposition temperatures over which smooth deposits can be maintained.

The factors influencing the cracking of the deposit are examined in detail. Internal stress can be influenced by the deposition conditions, particularly temperature, but under conditions of equal stress the incidence of cracking tends to be reduced when the Rh is deposited on a soft unstressed metal surface. Under typical deposition conditions it is shown that no cracking occurs up to 0.0002 to 0.0003 in., a range which incorporates the major proportion of industrial Rh plating. For thicker deposits cracking is reduced by lowering the internal stress, but in all known cases this can only be done at the expense of the surface smoothness of the deposit.

The importance of a correct treatment of the basis metal surface is emphasised, and techniques are described for Rh and Ag/Rh plating the most common of these.

The Electroplating of Palladium
E. R. THEWS, Metalloberflache, 1956, July, 193-196

A general review of the development, properties and uses of Pd as an electroplating material. It includes sections on the main processes used in obtaining a satisfactory deposit, and on the various types of plating baths.

LABORATORY APPARATUS AND TECHNIQUE

The Accurate Determination of Methane
B. DOMANSKI, A. JOURDAN and C. EYRAUD, Chim. anal., 1956, 38, Sep., 322-327

Describes an improved apparatus for the determination of methane based on the change in resistance of a Pt filament during the catalytic combustion of the gas. The apparatus has been adapted as a portable unit which is described in detail. Identical Pt-10%Rh filaments form two arms of a Wheatstone bridge circuit, one filament is used as the catalyst in the combustion of the gas to be determined which is passed over it in strictly regulated amounts, while the other is used as a standard resistance. The roles of the two filaments can be interchanged thus eliminating the inevitable drift in readings obtained by using only one filament. The apparatus has been used for two thousand measurements lasting a minute each, with great accuracy and sensitivity, without the filaments showing any sign of needing replacement.

The Behaviour of O$_2$ - H$_2$O$_2$ - H$_2$O on Bright and Platinised Pt
D. WINKELMANN, Z. Elektrochem., 1956, 60, (7), 731-740

Describes experiments on the electrochemical behaviour of H$_2$O$_2$; the cathodic reduction of O$_2$ and the oxidation and reduction of H$_2$O$_2$ on Pt were investigated. Experimental procedure and results are given, and reaction mechanisms postulated.

The Metallographic Preparation of Noble Metal Samples by Cutting with a Microtome
G. REINACHER, Z. Metallkunde, 1956, Sep., 607-613

Describes experiments on the preparation of slides of Ag, Au, Pd, Pt and some heterogeneous Ag alloys by cutting with a microtome instead of by the usual method of grinding with emery paper. The advantages of the method are enumerated.
A Simple and Inexpensive Large Area Rotating Pt Electrode
J. K. JONNNESSON, Chem. & Ind., 1956, Oct. 20, 1141-1142
Describes a very satisfactory large area rotating Pt electrode for use in amperometric titrations. It is prepared by coating a glass bulb blown at the end of a glass tube with a thin conducting film of Pt, electrical contact being made by means of a Pt wire sealed through the glass.

Precious Metal Coated Bombs for High Temperature Studies
The adaptation of stainless steel bombs for use with corrosive solutions at high temperatures by cladding with Pt or Au is described. Pt is used almost exclusively because of its superior mechanical properties and the fact that it can be soldered with pure Au. Full details are given of the cladding of a delta-sealed bomb and a smaller test-bomb with Pt, and also of the development of an Au-plated bomb for studying the recombination of hydrogen and oxygen formed in radiolysis in a reactor fuel solution. The limitations of the bombs due to the differing thermal expansion of stainless steel from those of Pt and Au are discussed, and a method of testing for defects in the precious metal coating is described.

METAL WORKING
Study of the Effect of Gases on the Melting, Casting and Working of Palladium
R. H. ATKINSON, T. Met., 1956, 8, No. 8, 1029-1035
Experiments were carried out on Pd and an alloy of 95.5% Pd, 4.5% Ru, to find the effect of oxidising, inert and reducing atmospheres on their melting, casting and working.
Several different linings were used for the crucibles in which the metals were melted, these were: proprietary MgO, alundum, zircon, ZrO2 and fused MgO and Al2O3. The gases used as deoxidisers in the melting of Pd were: H2, CO, city gas, C2H2, tank gas and commercial CH4. Gases other than reducing gases whose effects were tested were: N2, air, steam, He, A, O2 and CO2. In judging the quality of the metal after melting and working, the principal criteria were density, hardness and freedom from blisters after annealing.
Detailed tabulated results are given. It is shown that H2 has serious disadvantages as a deoxidiser since it tends to introduce impurities into the melt by attacking the crucible lining, thus giving very hard Pd. It can be used when the crucible lining is MgO or MgO cement, but it still tends to form a pitted surface because of the formation of steam at the surface causing the Pd to blister when annealed in air. These disadvantages are lessened by using the intermittent deoxidation technique in which the Pd or Pd alloy is melted in a He or A atmosphere and H2 is applied intermittently at intervals of a few seconds. CO is a satisfactory deoxidiser for Pd, but with the alloy some gas was retained in the metal giving rather low densities. All the other gases which were tried caused excessive blistering of the metal when annealed in air.
Air was found to be unsatisfactory as an annealing atmosphere since it contains free O2 which forms a subscale of oxide. H2 can be used but it gives a very hard metal which is difficult to work, as well as tending to aggravate any existing blisters; it is satisfactory, however, when diluted to about 10% with N2. CO also has disadvantages as an atmosphere because of the H2 present as impurity. The best annealing atmospheres were found to be: He, A, steam, CO2, N2 or N2/10%H2 (suitable for most industrial alloys).
The solubility of various gases, namely O2, air, N2, steam, CO2, He and A in molten Pd were investigated, and it was found that steam and O2 were very soluble, He appreciably so, and even A was soluble to some extent. The investigation of the diffusion of gases in solid Pd showed that the order of increasing rate was: inert gases <N2, CO2 <O2. In a separate experiment H2 was shown to diffuse about 10 times as fast as O2.

CATALYSIS
Studies of the Strength of Poison-to-Catalyst Bonds. Part I: Heats of Adsorption of Ethyl Sulphide and Thiophen on Platinum
It is known that near room temperature catalyst poisons are reversibly chemisorbed on to the catalyst surface, and in this paper the strength of the bonds involved is found by measuring the differential heat of adsorption for two typical poisons, ethyl sulphide and thiophen. The values obtained were 65-70 k. cal. per mole for ethyl sulphide and about 33 k. cal. for thiophen; the difference is presumed to be due to thiophen losing its resonance structure on adsorption. Both values are considerably greater than that for H2, 18-20 k. cal., as would be expected.
Catalytic Hydrogenation of m-Nitroacetylbenzenes to m-Alkylanilines
M-alkylanilines, previously only obtainable with difficulty, can be formed easily and in good yield by hydrogenation of m-nitroacetylbenzenes in the presence of palladium-black in glacial acetic acid with sulphuric acid monohydrate as activator.
Penex-Platforming of Naphthas
ANON. Petrol. Process., 1956, 11, No. 8, 66-67
Describes Universal Oil Products Company's recently developed Penex isomerisation process for increasing the octane content of naphthas. A fixed-bed Pt containing catalyst in a hydrogen-atmosphere is used; the catalyst does not need regeneration, and only a single heater and single reactor are needed. With recycle operation, product yields of around 99.5% can be obtained.

Electronic Theory of Catalysis (I) Metals
J. E. GERMAIN, Bull. Soc. chim. Fr., 1956, Parts 8-9, 1305-1313
A general review of the literature on the electronic theory of catalysis as applied to metals, from the point of view of electronic structure and chemisorption. The effect of alloying the metals, and of their texture, is discussed, as are catalytic supports and promoters. Pt and Pd are among the examples cited. (60 references.)

The Dehydrogenation of Ethane at Low Pressure on Incandescent Pt
Describes experiments carried out on the reaction of ethane on a Pt filament at temperatures between 1300 and 1800° K and from 10^-4 to 10^-7 mm. On a freshly prepared filament both ethylene and ethyl radicals are formed but after a short while the reaction stops due to poisoning of the catalyst by formation of a carbonaceous layer on its surface. The reaction proceeds again in the presence of water, or if the filament is heated in oxygen. The reaction mechanism and theoretical aspects of the phenomena observed are discussed in detail.

The Reactions of Ethylene with Deuterium over Various Types of Pt Catalyst
Describes experiments carried out on Pt pumice, platinised Pt foil, Pt-Al2O3, Pt-SiO2 and Pt-Al2O3-SiO2 to discover whether the kinetics of a typical heterogeneous reaction are affected by the type of catalyst used. Experiments were carried out on each catalyst to discover the course of the reaction at 0°C, and the effect of temperature and partial pressure variation. Detailed tabulated results are given which show little difference in the behaviour of the catalysts. The kinetics of the addition and exchange reactions are discussed.

Oxidation of Methane in a Homogeneous Phase by Air or “Active” Oxygen on Incandescent Pt
C. BYRAUD, B. DOMANSKI and P. BUSSEIRE, Compt. rend., 1956, 243, (13), 905-907
Experiments carried out indicate that the catalytic oxidation of methane over Pt takes place at least partially in the homogeneous phase by means of oxygen desorbed from the surface of the catalyst in an active state.

Heats of Adsorption of O2 on Ni, Pt and Ag
The adsorption of O2 is studied on powdered Ni, Pt and Ag samples in the temperature range 150-300°C and surface coverings 10^-4 to 10^-3. Results showed that the behaviour of Pt and Ag was very similar, but Ni showed certain differences. This suggests that the O2 chemisorption is not directly related to the electronic structure of the metals, providing a contrast to H2 adsorption.

Investigations on Catalysis XIV. Catalytic Oxidation of p-Cymene at Room Temperature
Experiments are carried out on the course and kinetics of the heterogeneous oxidation of p-cymene at room temperature using Pd-C, Pd-BaSO4 and CuO-BaSO4 catalysts. Detailed results are given in the form of graphs and tables. No oxidation is obtained with CuO-BaSO4 but Pd-C and Pd-BaSO4 were both found to be satisfactory catalysts.

CATHODIC PROTECTION
Platinum Anodes more Economical than Alloy Platinum-Palladium, NACE Task Group Concludes
NATIONAL ASSOCIATION OF CORROSION ENGINEERS, Corrosion, 1956, 12, Jul., 86
Describes the use of Pd-Pt alloys for testing as anodes in cathodic protection systems on the
hulls of ships. The lowest corrosion rate was recorded for the 90%Pt-Pd alloy, but since pure Pt was found to be more economical the use of the alloy was not recommended. Details of the properties of Pt anodes are given together with information on a new type of foil anode consisting of the alloy 80%Pt-Pd.

### ELECTRICAL ENGINEERING

**Selecting Materials for Electrical Contacts**

**Materials & Methods Manual No. 130**

V. G. Mooradian, *Mat. & Methods*, 1956, 44, Sep., 121-140

This manual tackles the problem of the choice of contact materials for various applications under the headings: evaluation of operating conditions, evaluation of life and reliability requirements, selection of basic contact material, and determination of contact design. Under the first two headings the mechanical and physical properties required from the material under various conditions are discussed. A chart of contact materials used for specific applications is given. The Pt metals feature prominently in this, they or their alloys being used among other things for contacts in thermostats, instruments, magnetos, motors and telephone equipment. The selection of the basic contact material is aided by a chart giving the advantages, disadvantages and applications of the common contact materials, alloys of Pt and Pd with Ir, Rh, Ru, Os and Au are resistant to oxidation at high temperatures and are capable of maintaining low contact resistance over long periods.

### ELECTRONICS AND TELECOMMUNICATIONS

**Thermionic Emission Constants of Ir**


The thermionic emission constants of polycrystalline Ir are measured with reasonable care, using thin ribbons of Ir as the emitting specimens. The values of the constants $A$ and $\phi$ obtained experimentally were:

- $A = 170 \text{ amp cm}^{-2} \text{ deg C}^{-2}$
- $\phi = 5.40 \text{ V}$

**Brazing Molybdenum and Tungsten Cathode Parts with Ruthenium**


In the development of thoria-coated indirectly heated molybdenum cathodes operating above 1500° C the problem arises of joining Mo and W. Several metals were proposed as brazing media and Ru was found to be the best, although Os and Ir are worthy of investigation. Brazing was carried out in 80% N2 and 20% H2 at 2100° C, the brazing medium consisting of Ru powder or a mixture of Ru and Mo powders. A eutectic melting at about 1900° C was formed between Ru and Mo allowing Ru to be used in a brazing operation below its normal melting point and safely below the melting point of Mo.

### TEMPERATURE MEASUREMENT

**Recent Developments in Temperature Measurement and Control**


An excellent critical review incorporating all recent developments of interest to the metallurgist. Summarised information on available methods of temperature measurement is given in tabular form. Brief reference to the standard 10 and 13% Rh-Pt:Pt thermocouples is followed by more details on special couples for high temperatures, including the 1%Rh-Pt:13%Rh-Pt, 5%Rh-Pt:20%Rh-Pt and 20%Rh-Pt:40%Rh-Pt. Protection of couples for use in liquid metal temperature measurement is also discussed. A section on gas-temperature measurement describes suction pyrometers in which the gas is drawn past the thermocouple. (28 references.)

The authors have developed a pneumatic pyrometer with venturi openings in place of orifices; this has many advantages over previous designs and is ideal for control work where a knowledge of the true temperature is unimportant. Instruments now being developed are described, including the suction pyrometer, total heat meter and pneumatic pyrometers. Most work has been done on the suction pyrometer and a new design now being marketed is described in which the Pt-metal thermocouple unit can be fitted with different heads for use under various conditions. The authors have developed a pneumatic pyrometer with venturi openings in place of orifices; this has many advantages over previous designs and is shown to have potentialities as a commercial continuous recording instrument. It has no upper temperature limit and a very quick response which give it advantages over the suction pyrometer. The difficulties encountered and the relative use of the various instruments in the temperature ranges <1000°C, 1000-1400°C, and >1400°C are described.
Suction Pyrometers in Theory and Practice

The factors to be considered in designing an efficient suction pyrometer are discussed. A commercially available instrument is described comprising a Pt:Pt-13%Rh thermocouple surrounded by radiation shields and incorporating devices for controlling and measuring the velocity of the gas. The optimum velocity is shown to be about 500 ft./sec. and the efficiency at various temperature levels is maintained by altering the number of radiation shields; it has been found advantageous to enclose these in a water-cooled jacket for use in open-hearth furnace uptake where there is a danger of overheating and slag attack.

NEW PATENTS

Reductive Alkylation of Organic Compounds
UNIVERSAL OIL PRODUCTS CO. British Patent 753,740
An organic compound having an amino or nitro group substituent is reacted, together with a ketone or aldehyde and hydrogen in the presence of a platinum-containing catalyst (not over 2% by weight Pt).

Dehydroisomerisation of Naphthenes
N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ British Patent 753,783
C₃ ring naphthenes are converted to the corresponding aromatic hydrocarbons by treatment, in the presence of hydrogen, at 420°C to 570°C, with a catalyst having a surface area greater than 300 sq. metres/gram and consisting of silica gel promoted with 0.1-0.8% alumina and 0.1-1% platinum.

Hydroforming
ESSO RESEARCH & ENGINEERING CO. British Patent 754,041
Hydroforming is carried out with the use of a fluidised desulphurisation catalyst which absorbs the sulphur removed and gives an H₂S-free naphtha product.

Hydroforming Catalyst
ESSO RESEARCH & ENGINEERING CO. British Patent 754,552
A hydroforming catalyst consists of 0.01-2% by weight of finely divided metallic platinum or 0.5-5% finely divided palladium supported on pure crystalline alumina. The alumina is prepared by heating beta alumina trihydrate to 750-1500°F. The catalyst is made by impregnating the crystalline alumina with a solution of a platinum or palladium salt, drying and calcining.

Recovery of Platinum from Catalytic Materials
AMERICAN CYANAMID CO. British Patent 755,487
Platinum-alumina catalytic material is digested with sulphuric acid. The alumina goes into solution and platinum remains in suspension. The platinum is flocculated and separated.

Cyclisation of Pentamethylenediamine
IMPERIAL CHEMICAL INDUSTRIES LTD. British Patent 755,534
Piperidine is made by contacting pentamethylenediamine in vapour state at elevated temperature (300-500°C) with a catalyst consisting of platinum (5%) on pellets of silica gel. An inert gas is preferably passed through the reaction zone.

Reforming of Gasoline Fractions
UNIVERSAL OIL PRODUCTS CO. British Patent 755,709
A gasoline fraction containing arsenic impurities is first freed of impurities by contact with an initial portion of a noble metal catalyst at 125-425°C and is then reformed during contact with another portion of the catalyst at 440-650°C. Catalyst comprising alumina, 0.1-8% of fluorine or chlorine and 0.1-1% platinum is referred to.

Electrical Contacts
JOHNSON, MATTHEY & CO. LTD. British Patent 756,393
A rivet-type electrical contact is made from a disc-like blank. A hollow shank is formed by extruding part of the material of the blank, the remaining unextruded part forming the contact operating face. The blank may consist of solid metal, such as platinum or palladium, or of bi-metal, i.e. a base metal backing, e.g. copper, and a precious metal facing layer.

Hydroforming
ESSO RESEARCH & ENGINEERING CO. British Patent 756,798
A hydroforming process in which the treated material, withdrawn from the hydroforming zone, is fractionated and the part boiling between 225°C and 300°C is recycled to the hydroforming zone. A platinum group metal-on-alumina catalyst is used, e.g. 0.05-5% platinum or 1.5-10% palladium by weight.

Platinum Catalysts
N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ British Patent 756,902
A platinum catalyst includes also a minor portion each of aluminium fluoride and aluminium.
A platinum group metal catalyst may be used in an electrolytic process of separating alkali metal ions from ions of zinc and/or aluminium.

Electrolytic Process
ROHM & HAAS CO. British Patent 757,928
Platinum electrodes are used in an electrolytic process of separating alkali metal ions from ions of zinc and/or aluminium.

Thermionic Valves
THE M-O VALVE CO. LTD. et al. British Patent 758,099
In a thermionic valve of the kind having a cathode comprising alkaline earth metal compounds in a molybdenum container, the active portion of the cathode, which consists of sintered tungsten, being welded to the molybdenum container, the inner surface of the container is coated with a layer of platinum, rhodium or iridium to prevent reaction of the alkaline earth metal compounds with the molybdenum during welding.

Thermionic Cathodes
THE GENERAL ELECTRIC CO. LTD. et al. See M-O VALVE CO. LTD. British Patent 758,099

Aromatisation and Naphtha Reforming
ESSO RESEARCH AND ENGINEERING CO. British Patent 758,680
A platinum group metal catalyst may be used in a method of converting aromatisable hydrocarbon material by contacting the material in the presence of added hydrogen at elevated temperature, withdrawing the contaminated catalyst, which is heated to burn off carbonaceous deposits and then passed to a purging zone for treatment with gas containing hydrogen and 45 vol.% of a gaseous hydrocarbon to remove water.

Spinning Nozzles
W. C. HERAEUS G.m.b.H. British Patent 758,507
A spinning nozzle is made of an alloy composed of 10–30% iridium and remainder platinum. Up to 5% of other platinum group metals may be included, preferably rhodium and palladium.

Purifying Acrylonitrile
MONTECATINI SOC. GEN. PER L’INDUSTRIA MINERARIA E CHIMICA British Patent 758,577
A supported platinum or palladium catalyst is used in a process of purifying acrylonitrile containing acetylene polymers by hydrogenation at temperatures of 0–50°C in a hydrogen atmosphere and a pressure of 1–20 Atm, pure acrylonitrile being separated from the product by fractional distillation.

Welding Contacts to Wires
WESTERN ELECTRIC CO. INC. British Patent 759,076
Discloses apparatus for welding contacts on to the ends of wires mounted in a row on an electrical relay component. The contacts may be made from contact tapes of composite material, e.g. a body of nickel-silver coated with a thin layer of palladium either on the upper surface, the under surface or both upper and under surfaces.

Analytical Electrodes
JOHNSON, MATTHEY & CO. LTD. British Patent 759,288
An electrode for electrochemical analysis of the kind comprising a perforated cylindrical or flat electrode member mounted in a cylindrical or rectangular metallic frame, has the electrode formed of expanded metal, preferably of platinum or other platinum group metal or alloy.

Catalytic Conversion of Hydrocarbon Oils
N.V. DE BATAAFSCHTE PETROLEUM MAATSCHAPPIJ British Patent 759,310
In the conversion of a hydrocarbon oil in vapour phase using a long narrow reaction zone and a separate catalyst regeneration zone, regenerated catalyst is introduced into the vapours near the inlet end of the reaction zone and immediately. Suspended catalyst is separated at a point between the positions and at a point near the outlet end. A supported platinum catalyst may be used.

Hot Air Generator
GAZ ET CHALEUR British Patent 759,520
Air heating apparatus comprises a heating chamber having a catalytic combustion heating...
element and an air flow duct conveying the air past the wall of the chamber so that the air is heated by indirect heat exchange. The heating element consists of a grid or other perforated member provided with a platinum catalyst.

**Grid for Electric Discharge Tubes**

STANDARD TELEPHONES & CABLES LTD. *British Patent 759,552*

A metal grid is made by winding a support frame with a jacketed metal wire and then removing the jacket from part at least of each turn of the winding. The wire may be Wollaston wire with a core of gold, platinum or aluminium and a jacket of silver. Jacket removed chemically or electrolytically.

**Heat Treatment of Metal Catalysts**

G. STETTER *British Patent 760,413*

Two-dimensional, net-like catalysts of platinum or platinum alloys, e.g. platinum-rhodium catalysts as used for ammonia oxidation, are given a microcrystalline structure by heating the catalyst by passage of electric current through it while protecting it from heat losses by conduction or radiation by placing the catalyst between heat insulating masses (fireproof metal oxides or asbestos) adjacent to, but not in contact with, it. The surfaces of the insulating masses may be coated with platinum.

**Hydroforming**

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent 2,746,909*

The invention concerns a method of hydroforming naphtha with use of a platinum group metal catalyst and is of the regenerative type. The hydroforming zone is operated at about 200 lb. pressure.

**Preparing Platinum-Alumina Catalysts**

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent 2,746,937*

Platinum-alumina or palladium-alumina catalysts are made by mixing platinum or palladium with an alumina hydrosol prepared by treating aluminium metal with a dilute acid in presence of a catalytic amount of mercury and drying and calcining the product.

**Hydrogenation of Dicyanobutene**

E.L. DU PONT DE NEMOURS & CO. *U.S. Patent 2,749,359*

Relates to production of adiponitrile in a hydrogenation reaction employing a palladium-on-coconut-charcoal catalyst. A gaseous mixture of 1,4 dicyanobutene and 10-100 mols of hydrogen per mol of the material is passed at a pressure of 0.5-5 atmospheres at 200-350°C into contact with the catalyst.

**Reforming Catalysts**

ATLANTIC REFINING CO. *U.S. Patent 2,750,329*

A reforming catalyst is made by contacting a cracking component silica and zirconia, magnesia, alumina and/or thoria with an aqueous solution of a platinum or palladium compound, subjecting the component and solution with constant moisture content to a temperature and for a time sufficient to fix at least 50% of the metal deposited on the component, drying and reducing the metal compound to the metal.

**Reforming Catalyst**

HOUDRY PROCESS CORP. *U.S. Patent 2,751,333*

A dehydrogenation and isomerisation catalyst is composed of 0.05-2% by weight platinum on an activated alumina which has been acid treated with an organic carboxylic acid.

**Low Pressure Hydrogenation**

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent 2,757,128*

The invention concerns the treatment of gasoline hydrocarbons to improve their lead susceptibility, their stability and to reduce the sulphur content of raw gasoline fractions. The material is fed together with hydrogen to a zone containing a fluidised bed of hydrogenation-dehydrogenation catalyst (platinum-on-active alumina and containing some HF).

**Hydroforming Process**

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent 2,758,062*

In a two-stage hydroforming process for hydrocarbon fractions, a platinum- or palladium-on-alumina catalyst is employed in the first stage and a Group VI metal oxide or sulphide catalyst in the second stage.

**Regeneration of Hydroforming Catalysts**

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent 2,758,063*

A continuous hydroforming process using a platinum-containing catalyst is effected by passing hydrocarbon vapours and hydrogen-containing gas through a bed of finely divided platinum-containing alumina particles at a pressure such that coke or carbonaceous material is deposited. The particles are treated with a hydrogen-rich recycle gas free of C4 and higher hydrocarbon at hydroforming pressure and temperature long enough to remove the coke or carbonaceous material and thus reactivate the catalyst, which is then reused.

**Reforming of Gasoline Fractions**

UNIVERSAL OIL PRODUCTS CO. *U.S. Patent 2,758,064*

In a reforming process using a catalyst comprising platinum, alumina and combined halogen, in which one or more of the catalyst components is
deactivated by excess sulphur and nitrogen, such deactivation is prevented by hydrogenating and hydrocracking the charge in the presence of hydrogen and a hydrogenating catalyst and converting the sulphur and nitrogen contaminants into hydrogen sulphide and ammonia, which can be separated and removed.

Conductors
E.I. DU PONT DE NEMOURS & Co. U.S. Patent 2,758,267
An electric device comprises a silver conductor element of positive polarity and a conductor of negative polarity on which silver can deposit. Both conductors are arranged on a dielectric with a barrier composed of gold, platinum or palladium on the dielectric between the conductors and spaced from the negative polarity conductor.

Hydrocarbon Conversion Catalysts
UNIVERSAL OIL PRODUCTS Co. U.S. Patent 2,759,897
A catalyst is made by commingling a solution of a platinum compound with alumina in amount to form a catalyst containing 0.01-10% platinum and calcining the composition at 700-1100°F in an atmosphere of air containing at least 0.01578 lb. of water per lb. of dry air.

Production of Hydrogen and Carbon
HOUDRY PROCESS CORP. U.S. Patent 2,760,847
In the production of hydrogen and carbon from gaseous hydrocarbons by contacting the charge at 1200-2000°F with an atomised stream of molten metal contact material, the contact material contains 1-10% by weight of the molten metal of a dehydrogenisation catalyst, e.g. of platinum.

Catalyst Reactions
E. P. SCHWARZENBECK U.S. Patent 2,760,912
A naphtha fraction is contacted under suitable reforming conditions with a catalyst composed of platinum or palladium supported on a porous carrier. The catalyst is made by combining a hydrous carrier material, an ammine complex of platinum or palladium and a water-soluble promoting agent (alcohol or ketone) and heating the mixture to decompose the compound and leave a metal residue on the porous carrier.

Composite Metal Rod
BAKER & CO. INC. U.S. Patent 2,761,207
A composite metal rod is made by anodising a refractory core and telescoping it into a platinum metal sheath, the inner surface of which has been etched. A portion of the sheath extends beyond the core and is gripped for snug-fitting the sheath to the core. The extension is removed and the composite rod hot swaged with progressive reduction of cross-sectional area under progressively reduced temperatures.

Catalytic Conversion of Hydrocarbons
ESSEO RESEARCH & ENGINEERING CO. U.S. Patent 2,761,819
A hydrocarbon is contacted at 850-1000°F in the presence of hydrogen to convert it to a higher octane number with a platinum-alumina catalyst which has previously been treated with hydrogen at 1000-1300°F for 2-24 hours.

Platinum Catalyst
SHELL DEVELOPMENT CO. U.S. Patent 2,762,781
A catalyst consists of an inert carrier incorporating 0.05-1% platinum with 0.03-0.4 mole per kilogram each of aluminium fluoride and aluminium borofluoride as acidic promoters.

Silica-Alumina Catalyst
UNIVERSAL OIL PRODUCTS Co. U.S. Patent 2,763,623
A synthetic silica-alumina cracking component is heated at 900-1800°F in presence of steam until the cracking activity is reduced below 50% of the original. Platinum, 0.01-2.5% by weight, is then composited with the product.

Catalyst Regeneration
ESSEO RESEARCH & ENGINEERING CO. U.S. Patent 2,764,528
A spent platinum-alumina hydroforming catalyst is regenerated by suspending it in a high boiling naphthenic oil, hydrogenating the suspension for 10 secs. to 10 mins. at a hydrogen partial pressure of 1,000-10,000 lb./sq. in. at 600-900°F, replacing the oil, after settling, by washing the suspension with an equal volume of a low boiling saturated wash oil in a hydrogen atmosphere at below 500°F and removing the oil by drying in a hot hydrogen atmosphere.