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ITEM No. 21

THE PLATINUM METALS INDUSTRY IN GERMANY

LONDON—H.M. STATIONERY OFFICE

THE PLATINUM METALS INDUSTRY

IN GERMANY

Reported by:

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METALLURGY.

British Intelligence Objectives Sub-Committee.

32 Bryanston Square, W.1.

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INTRODUCTION

On the industrial side, the greater part of the work of the investigators lay with the two main German platinum metal firms, viz:- W.C. Heraeus and Siebert-Degussa, both of Hanau. The severe war damage to almost all the plants of these concerns with the reputed loss of documents by fire and the confiscation of products by the Occupying Forces, made interrogation of personnel the most vital channel of technical intelligence. In this connection, it was felt that the visit of the Team was a little belated, because the 'targets' had been examined previously by investigators on other matters and the Germans were always ready with a "drill" and even typescript notes on processes, etc. Nevertheless, it was generally found that mutual appreciation of specialist knowledge eventually produced the information that was sought.

Of the subsidiary 'targets', the visits to the Institut (Laboratorium) für Physikalische Chemie und Elektrochemie der Technische Hochschüle, Wiederholdstrasse 15, Stuttgart and the Forschungsinstitut für Edelmetalle, Schwäbisch-Gmünd were specially valuable in providing information on the fundamental researches on precious metal themes that have been carried out in Germany since 1939.

CONCLUSIONS

1. Whilst the broad picture of platinum metal activities in Germany has much in common with the general situation in the United Kingdom, there have taken place in the Reich, significant additions to both fundamental knowledge and processing techniques. At the same time, the Germans appeared unaware of several developments in the West which are playing vital parts in the commercial use of platinum metals.

2. A high level of research activity on precious metal problems has been maintained in Germany during the war period. This is largely attributable to platinum metal shortage and the consequent desire to find alternative or more economical materials.

3. The most important recent advances made by the German platinum metal industry may be summarized as follows:-

(a) High frequency vacuum melting and casting of alloys is regular practice where difficulties might otherwise be encountered due to the presence of gas or of a readily oxidizable constituent in the melt.

(b) Special attention has been directed to the production of optically perfect mirror surfaces by the evaporation of thin metal films on to glass. In particular, the production of highly reflecting rhodium mirrors in the largest sizes required by industry had become normal commercial practice.

(c) In collaboration with the industry, an unexpectedly large number of fundamental investigations had been carried out by German scientists on precious metal themes. In particular, reference is made to new studies on platinum-gold alloys whose proper heat treatment is a matter of considerable industrial importance.

(d) Attention has been directed to the effect of small quantities of beryllium as a potent hardener for platinum, and whilst some very interesting results have been obtained, opinion is divided on the technical suitability of such alloys.

(e) Very substantial progress has been made in the macro-quantitative spectrographic analysis of binary platinum alloys and techniques permit in several instances the determination of up to 10% of an alloying addition with good accuracy.

(f) The production of refractory crucibles, tubes, etc. for the precious metal industry has been maintained at the highest quality level and it seems doubtful whether similar materials available in the United Kingdom or the States bear comparison.

4. Information has been collected on certain refining, analytical and production techniques used by the platinum metals industry in Germany. This contains certain novel features which may prove valuable to home platinum metal producers and users.

INDIVIDUAL FIRMS, TECHNICAL INSTITUTES, ETC.

In the subsequent pages, there is summarized intelligence obtained from the German 'targets' visited. In several instances, more comprehensive details on processes, techniques, etc. were prepared by German personnel at the request of the Team. These are collected together in appropriate appendices.

W.C. HERAEUS, HANAU.

DATES VISITED

November 19th, November 20th and November 30th, 1945.

PERSONS INTERVIEWED

Dr. Heraeus (junior) - Director
Dr. Canthal - Director
Dr. K. Ruthardt - formerly head of the Research Department, now secretary to Dr. Canthal.
Dr. P. Haas - Chief Chemist
Herr Ackman - Engineer

Arrangements were put in hand for the evacuation of Dr. K. Ruthardt to England for more intensive interrogation.

It was explained that Dr. Heraeus (senior) was not available as he had been "removed" for his earlier Nazi activities.

CONDITION OF PLANT

The Precious Metal Refinery, the Laboratories and the rhodium evaporation plant are wrecked. The melting shop which is equipped inter-alia with four point high-frequency melting equipment including one vacuum outfit, is intact. The rolling mills and ancillary equipment have been brought back from their evacuation to Erlangen and this modern plant was in process of re-installation.

Notwithstanding the severe damage, a determined effort was in progress to get going again, even though significant quantities of materials were lacking - "frozen" by the Occupying Forces. The "new refinery" was comparable with an ordinary elementary chemical laborator

SOURCES OF PRECIOUS METALS

In normal times, Heraeus obtained their pure metals by direc purchases and by the refining of scrap.

SPECTROGRAPHIC QUALITY CONTROL

In collaboration with Siebert-Degussa, Heraeus have in recent years devoted a great deal of attention to spectrographic assay and quality control of platinum metals. The work of Dr. K. Ruthardt and his colleagues is outstanding in this connection. In general, arc spectra were used for qualitative and quantitative "trace" determinatic whilst for "macro quantitative work" spark spectra were used with the electrodes of the alloy under investigation in the form of wire. Fc binary alloys of platinum, it was possible in certain cases to determine up to 10% of an alloying addition. Thus, rhodium could be estimated to the nearest half per cent in the alloys containing up to 10% of the element. For "trace" work, it was preferred to use a strong current in the arc circuit, viz:- 25 amperes, and short exposure times. By this means it had been possible to determine with accuracy 0.1% of iridium in platinum and there were hopes of determining as little as 0.01 to 0.02% of iridium. Experiments had been carried out with an interrupted arc and the results were just beginning to show promise when the apparatus was destroyed. For qualitative work, some lines found to be most sensitive in the presence of platinum were:-

Iridium..........3220 and 2919 (?)
Rhodium..........2400 (?), 2500 (?), 2508 and 2520

A supplement to the above, prepared by Dr. K. Ruthardt is contained in Appendix 1.

QUALITY CONTROL OF PLATINUM FOR RESISTANCE THERMOMETERS

This is always carried out by temperature co-efficient of resistance measurements. For resistance thermometers, the minimum permissible value of α determined on the platinum wire used, was 3.87×10^{-3} and after assembly of the thermometer the figure fell to $3.85\ (\pm 0.005) \times 10^{-3}$. The best material ever made, had $\alpha = 3.91 \times 10^{-3}$. Heraeus were hopeful of getting into production again with platinum resistance thermometers for the United States Forces. The values of α recorded indicate that Heraeus material is inferior to that available commercially in England.

PROCESSING TECHNIQUES

Heraeus favour high frequency melting of commercial heats and are well equipped to this end. Melts which hold on to gas or tend to oxidize can be melted and cast in vacuum. Charges are normal - 100 ounces Troy.

Thermo-element platinum is melted in air in pure lime crucibles. Magnesite crucibles are preferred for alloys such as tungsten-platinum. Carbon dioxide has been found to be a good protective atmosphere for 8.5% nickel-91.5% platinum and 5% tungsten-95% platinum alloys. Palladium alloys are usually melted and cast in vacuo to obviate gas troubles and beryllium-platinum alloys are also vacuum melted to prevent oxidation of the beryllium. Hard alloys such as those used for pen points were manipulated by melting the constituent metals together in a zirconia crucible, allowing the heat to freeze and then breaking up the ingot. The alloys were of too high a melting point to cast successfully, even if this was desirable.

A demonstration was given of the vacuum melting of the alloy 32% silver-68% gold - platinum metals were not available, but the procedure followed normal practice. An alumina crucible and a graphite mould were used. These, complete with high frequency coil and auxiliary fittings were contained in a copper vacuum pan approximately two feet diameter and twenty inches high. A quartz window permitted the melting and casting process to be viewed with ease. Vacuum tightness of the apparatus was secured through a special quality rubber gasket - between the lid and pan proper, fastening being at eight points. Evacuation was by Pfeiffer rotary oil pump without diffusion pump. Detailed procedure was as follows:-

The metals were fed into the crucible and "run down" in air. The lid of the vacuum pot was clamped into position and the apparatus pumped out to a pressure of approximately 0.5 mms. of mercury. This took about half an hour. The charge was then reheated and after melting, gas evolution proceeded normally with a pressure rise to about 2.0 to 3.0 mms. of mercury. When there was no further evolution of gas, casting was carried out by manual tipping of the whole apparatus - the high frequency current being cut off. The melt transferred to the mould cleanly and without splashing and the resulting ingot was of close texture and showed normal shrinkage.

When casting alloys susceptible to segregation and severe coring, due, say, to a wide range between liquidus and solidus temperatures, Heraeus preferred to use a mould slightly tapered from top to bottom and which has a small heating coil around the top. Such an arrangement ensures relatively slow, uniform cooling of the casting and adequate feeding.

Heraeus obtain all their refractory materials from Siebert-Degussa.

SPECIAL ALLOY COMPOSITIONS

Catalysts

For the gauze catalysts used in the oxidation of ammonia, Heraeus made 5% rhodium-platinum alloy under Baker patents. Suppli of rhodium were inadequate to permit the more satisfactory 10% rhodium level. During the war period the tendency had been in Germany to favour the use of atmospheric rather than high pressure plants so as t keep catalyst losses to a minimum. A cobalt oxide catalyst for use in conjunction with platinum metals had been developed for operation i atmospheric pressure plants.

Spinnerets

For spinnerets, the preferred compositions were:-

(a) 25.0% platinum-74.5% gold-0.5% rhodium and
(b) 40.0% platinum-59.8% gold-0.2% rhodium

The effect of the small rhodium addition to these alloys was to cause grain refining and enhancement of workability.

Miscellaneous

Beryllium hardened platinum alloys had been investigated, but notwithstanding the fact that Heraeus have a patent, they had not exploited them commercially. The difficulties were stated to be:-

(a) Making alloys of predetermined compositions containing 0.01-0.1% beryllium.
(b) Impairment of corrosion resistance when beryllium is added to platinum.

For electrical contacts carrying small currents, a whole range of palladium-gold alloys had been used.

PRODUCTION OF MIRRORS BY RHODIUM EVAPORATION

As a result of the investigations carried out at the Heraeus-Aürwärter Laboratories at Onstmettingen, the production of the largest sizes of mirrors by evaporation of rhodium on to glass had been accomplished. The Hanau Works had contained fine production plant for this work, but most of it had been destroyed by bombing. In the process, it was important to use rhodium of the highest purity in the form of wire 0.3 mms. diameter.

INDEPENDENT LABORATORIES OF DR. HERAEUS (SENIOR) AND DR. AÜRWÄRTER, ONSTMETTINGEN

DATE VISITED

November 22nd, 1945.

PERSONS INTERVIEWED

Dr. Aürwärter - Joint Director
Dr. Heisinger - Scientist

These Laboratories were originally situated at Hanau, but had been evacuated to the remote mountain village of Onstmettingen. They constitute a private enterprise run by Dr. Heraeus (senior) and Dr. Aürwärter. Almost the whole of the research effort was concerned with the physical, mechanical and electrical properties of surfaces and especially those obtained by applying very thin films of metal on to glass. An important application was the perfection of strong, abrasion resistant films for corrosion resistant mirrors, and Dr. Aürwärter and his colleagues had been directly responsible for the development of the rhodium mirror production plants at the Hanau Works of Heraeus.

Metal evaporation was the most favoured production technique and by this method satisfactory mirrors up to 40 cms. in diameter could be produced. The apparatus was subjected to brief inspection and appeared conventional in character.

Studies had been directed to the properties of other metal surfaces, and most promising results were being obtained with aluminium, though this metal lacked the high hardness of rhodium. However, its reflectivity was better, viz:-

Wave length of light A°	% total reflection - rhodium	% total reflection - aluminium
2000	67	83
3000	67	86
5600	67	90.5
8000	67	88

The thickness of the metal films was measured by a conventional wave length of light formula. It was learned that during recent years aluminium mirrors have been supplied to almost the whole of the German industry requiring mirrors for epidiascopes, etc. At present, the Laboratories were making mirrors for the American Army.

It was also learned that there was evacuated from Hanau to these Laboratories for safety some 800 kilogrammes of platinum metals which had

SIEBERT-DEGUSSA, GOLD AND SILVER REFINERY, 2-15, GUTLEUTSTRASSE, FRANKFURT.

DATE VISITED

November 19th, 1945.

PERSONS INTERVIEWED

Dr. Truthe - Manager
Herr Weichel - Development man
Frau Martin - Secretary to Truthe

SILVER REFINING

The plant is intact.

The refining of silver is normally carried out by the Moebius electrolytic process using anodes of 98 to 99% purity made by the concentration of scrap. Each unit cell in the plant produces 25 kilos of fine silver per day and the total capacity comprised approximately 72 cells in banks of six. The silver crystals falling from the cathodes were caught on metallic false bottoms in the cells which were periodically removed and the fine silver crystals centrifuged free from surplus electrolyte. It was stated that slimes were not troublesome in the process and when they accumulated were also removed by centrifuging. There was no experience of refining silver alloys with high contents of copper, nickel, zinc, etc.

GOLD REFINING

The electrolytic gold refining plant was of the conventional Wohlwill type. The gold cathode crystals were freed from electrolyte by centrifuging.

Platinum metal residues accumulated in the course of the silver and gold refining processes were concentrated and shipped to the Hanau Works for separation and purification.

More comprehensive information on the gold and silver refining processes is contained in Appendix 2.

DENTAL ACTIVITIES

A small, well equipped dental section was in mild operation and it was explained that silver and gold plated cast dentures respectively, in a basis alloy of silver-copper-cadmium, were being made as a substitute for the proprietary "Palliag" composition which had not been produced during the war period.

SIEBERT-DEGUSSA PLATINUM WORKS
13 HAUPTBAHNHOFSTRASSE, HANAU.

DATES VISITED

November 19th, November 29th and December 3rd, 1945.

PERSON INTERVIEWED

Dr. K.W. Fröhlich - Acting Manager

The previous Managers, viz: Herr Hans Siebert and Herr Wilhelm Duchardt had been "removed".

CONDITION OF THE PLANT

The Platinum Metal Refinery and the Laboratories appear to be irretrievably wrecked. The melting shop was damaged but repairable whilst the rolling mills and associated equipment were undamaged and mildly active.

SOURCES OF PRECIOUS METALS

The main source of refined platinum metals was slimes, etc. from the Frankfurt Gold and Silver Refinery plant and high quality scrap. Purchases of pure metals were also made from Russia prior to hostilities.

REFINING METHODS

A summary prepared by Dr. Fröhlich is contained in Appendix 2.

GENERAL ANALYTICAL TECHNIQUES

The reputed loss of records by enemy action rendered it impossible to provide absolutely exact data, but Dr. Fröhlich indicated that the separation procedures for the platinum metals used by Degussa followed closely on the commercial refining techniques.

SPECTROGRAPHIC ANALYSIS

It was explained that Degussa had collaborated closely with Heraeus in the development of macro quantitative spectrographic assays for platinum metals. The urge behind this work was to limit eventually the number of time consuming wet assays to a minimum. The apparatus used in the work had consisted of a Zeiss Q. 24 high resolution spectrograph with a similar microphotometer and Fröhlich was convinced that the finer points of the technique were wholly due to the "know how" of the technician. For macro quantitative work, spark spectra were used whilst for micro quantitative and qualitative work arc or spark spectra were used. When the investigations had terminated, methods had been worked out for determining in platinum, rhodium up to 5%, iridium up to 7% and ruthenium up to 10%. The macro side of palladium determinations had not been studied, but at the micro end, as little as 0.01% of palladium in platinum could be identified.

QUALITY OF PLATINUM FOR THERMOMETRY

Control was always by temperature co-efficient of resistance measurements and the value of α for regular production quality ran 3.90×10^{-3} to 3.91×10^{-3}. One batch had been made having $\alpha = 3.925 \times 10^{-3}$. Secondary control was by spectrographic examination of the finished metal.

PROCESSING TECHNIQUES

The melting of pure platinum was always carried out in air in lime crucibles. These latter were made at the Hanau Works from blocks consisting of pure lime and 5 to 10% of egg shells thoroughly mixed with it. The powder was pressed at 20 kilogrammes per square cm., dried, but not fired. If there was significant delay in using the pressings then they were stored in vacuum. For alloy heats, alumina, zirconia and zircon crucibles were used and these were purchased from Chemfer of Eisenach or the Degussa Refractory Plant.

Whilst a small amount of platinum metal melting was carried out by gas fusion, (hydrogen and oxygen; - city gas and oxygen were always avoided) all important melts were made by high frequency heating. The high frequency plant was only mildly damaged. A typical charge of platinum metals would weigh 10 kilogrammes and would be melted in a crucible 8 cms. in diameter.

"Undressed" iron moulds were used for platinum and its alloys. For alloys containing a readily oxidizable constituent or susceptible to gas troubles, high frequency vacuum melting and casting was practiced. For this, the assembly comprised a plastic or resin box 18" long, 10" wide and 10" deep lined inside with asbestolite. The vacuum joint to the lid was of rubber and the top contained a quartz inspection window. The box contained the high frequency coils, mould and necessary connections. The vacuum technique had been found satisfactory for making alloys of controlled composition, particularly those of the tungsten-platinum type.

SPECIAL ALLOY COMPOSITIONS

Electrical Contacts

Nickel-platinum alloys containing up to 10% nickel have been used as contact materials and in the processing of these, manganese is added as a deoxidizer.

5% tungsten-platinum alloy was used for electrical contacts.

Beryllium-platinum alloys

Much attention has been given by Degussa to the effect of small additions of beryllium as a potent hardener to platinum and paten have been taken out and the results also published in the literature (these are already available in U.K.). Not much more than 0.1% beryllium sufficed for most applications. The glass industry was using platinum containing 0.07% beryllium and preferred it to pure platinum (in this connection Degussa supplied only "blanks" to the glass fibre manufacturers who fabricated their own bushings - this ties up with U.K. practice). Beryllium-platinum alloys also found use as contact materials.

Crucibles

Degussa had supplied large crucibles in the 3% rhodium-platir alloy to Zeiss. The use to which they were to be put had not been disclosed by the purchaser - but the melting of optical glass was a goc guess. Laboratory crucibles in the platinum alloy containing 0.3% iridium and 1.5% rhodium were sold more on the basis of the higher pri obtained than because of technical merit.

Spinnerets

For spinnerets, Degussa preferred 50% platinum-50% gold to the more usual 70% gold-30% platinum composition.

Clad materials

The potentialities of clad metals had not got beyond preliminary Laboratory investigation, but silver coated iron for the chemical industry was regular production and a digest of the situation is given in Appendix 2.

Very small quantities of platinum-cobalt alloys had been mad converted into permanent magnets and used in amplifiers for the deaf.

SIEBERT-DEGUSSA REFRACTORY MANUFACTURING PLANT STIERSTADT, Nr. FRANKFURT.

DATE VISITED

November 20th, 1945.

PERSON INTERVIEWED

Dr. Stapelfeldt - Chief Chemist

This works was originally located in Frankfurt, had been temporarily evacuated to its present site (a disused aluminium bronze works) and was ready for return to Frankfurt. The plant was intact and ready to go into production when gas was available.

It was explained that the factory manufactured all classes of crucibles, tubes, etc. for high temperature service. The maximum size crucible normally made had a top diameter of 120 mms. and was of similar height. Most of the raw materials for the plant were purchased from Martins werk, Bergheim. The impurities present in the oxides purchased were normally not in excess of 0.5% and usually comprised silica, iron oxide and rare earth oxides.

ALUMINA ARTICLES

A 500 kilo charge of the oxide was ball milled to particles of approximately 5μ. The material was extracted with hot hydrochloric acid and washed free from any iron present. It was dried, and a slurry made consisting of equal parts of the oxide and water. 0.5% magnesium fluoride and 0.5% hydrochloric acid were then incorporated in the slurry and the crucibles or tubes slip cast in plaster moulds. Drying was carried out for from four to six hours at 100 to 120°C, and the refractory articles were then fired by slowly raising them to a temperature of 1900°C and holding them at this temperature for between one and four hours, depending on the size of the articles. Two to three hours was normal with a total heating time of ten to fourteen hours.

BERYLLIA ARTICLES

The treatment of the raw materials was substantially the same as for the alumina (above) but no additions were made to the slurry.

MAGNESIA ARTICLES

The raw material was cleaned and ball milled as in the case of alumina, but these crucibles, etc., could not be slip cast and they were "tamped" with 2 to 3% of magnesium chloride or hydrochloric acid as a binder. The final firing of magnesia refractories was at 1600°C with the same slow heating up cycle as for alumina.

No information could be obtained on the manipulation of zirconia or zircon as Herr Ahreus, the expert, was not available.

On the subject of the maximum temperatures at which various refractory articles might be used without slagging, Dr. Stapelfeldt gave the following figures:-

Alumina	1900°C
Zirconia	2000°C
Thoria	2100°C
Beryllia	?

As regards resistance to thermal shock, Stapelfeldt thought that beryllia was best, (better than zircon), then came alumina and finally spinel. Stapelfeldt suggested zirconia as the preferred material for platinum melting.

THE KAISER-WILHELM INSTITUT
evacuated to:-
INSTITUT (LABORATORIUM) FÜR PHYSIKALISCHE CHEMIE UND ELEKTROCHEMIE DER TECHNISCHE HOCHSCHULE, 15, WIEDERHOLDSTRASSE, STUTTGART.

DATE VISITED

November 23rd, 1945.

PERSONS INTERVIEWED

Dr. O. Kubaschewski - Deputy Director
Dr. G. Schrag - Research worker - part of time.

It was stated that the Director, Dr. G. Grubbe was not available as he had been held by the Americans for some two months, though for non-political reasons.

The investigations were originally carried out at the Kaiser-Wilhelm Institut, but this had had to be vacated due to war damage. Moreover, even the present facilities have not proved satisfactory and certain of the workers on precious metal themes had transferred to the Forschungsinstitut für Edelmetalle, Schwäbisch-Gmünd.

It was explained that the Institut was concerned wholly with investigations of a fundamental character though the themes were suggested by commercially interested firms such as Heraeus and by the Air Ministry of the Reich Government. It was certain that the limited quantities of precious metals available in Germany during the war period had stimulated a number of researches in the hope of finding more economical substitutes.

Dr. Kubaschewski provided a full written "Summary" of the activities of his Department during the last six years together with reprints of most of the publications of precious metal interest. Appendix 3 gives extracts from the "Summary" dealing with precious metal matters and schedules all the reprints provided. Among the more important results obtained, the following merit special comment:-

(a) Re-investigation of the binary silver-platinum, nickel-platinum, copper-platinum and nickel-gold systems. The work on the first three of these was complete, whilst that on nickel-gold had yet to be finished. One reason for carrying out this work was that earlier workers did not appear to have given adequate anneals to their samples and consequently had not been investigating "equilibrium conditions".

(b) The order-disorder transformations in the copper-gold system had been re-investigated. A special technique had been evolved, whereby alloy samples were used as catalysts for the decomposition of formic acid vapour. Thus the catalytic activity of a specimen would be measured after a heat treatment calculated to bring about a solid-solid transformation.

(c) The gold-iridium alloys had been examined for age-hardening characteristics.

(d) At the request of Heraeus an investigation had been carried out to determine why certain binary alloys of gold with base metals at elevated temperatures, e.g. 930°C oxidize to the same extent as corresponding platinum alloys under similar conditions. The approach in this case had been to calculate the free energy change for the formation of the oxide of the less noble metal. In the course of this work it had been shown experimentally that the mechanism of oxidation of platinum-rich, nickel-platinum alloys was the same as that of pure nickel.

(d) The volatility of platinum in air at elevated temperatures had been re-investigated and equilibrium constant measurements had tentatively established the formation of PtO_2, PtO_3 and PtO_4. Platinum loss in the presence of oxygen accordingly takes place via the oxide phase.

FORSCHUNGSINSTITUT FÜR EDELMETALLE, SCHWÄBISCH-GMÜND

DATE VISITED

November 24th, 1945.

PERSONS INTERVIEWED

Dr. E. Raub - Director
Dr. Esch - Investigator

The premises of the Institute were quite intact and normal progress of activities appeared to be held up due only to the sporad availability of utilities. Dr. Raub confirmed that certain member of the Institut (Laboratorium) für Physikalische Chemie und Elektro der Technische Hochschule, Stuttgart, had been working at Gmünd duri the war period, due to the destruction of their own Laboratories.

The scope and quality of the equipment for metallurgical investigation was impressive and it would seem to have been the pol of the Institute to discard obsolete equipment as soon as up-to-dat apparatus and instruments became available.

Dr. Raub explained that the general situation at Gmünd in relation to researches was substantially the same as that at the Institut at Stuttgart, viz: fundamental problems were investigated the request of industry or the Government. A considerable volum of academic work on these lines had been undertaken and as far as possible Raub provided reprints of published papers. These are scheduled in Appendix 4. In this connection the following merit special comment:-

(a) The investigations on the binary platinum-copper, platinum-nickel, and platinum-silver alloys originally initiated at Stuttgart had been finished and published at Gmünd.

(b) Dr. Esch had re-investigated the platinum-gold system put forward a new diagram. The work was unpublished. The tentative form of the diagram is contained in Appendix 4.

(c) The binary alloys of gold, silver and copper with the zirconium and titanium had been studied and the results published than on the zirconium alloys - which was actually finished - and th on the gold-titanium alloys which was incomplete.

(d) The super lattices in the gold-copper system had been re-investigated including the influence of nickel on this system. had been found that early American work and that of Bunn in German were unreliable because alloy samples "in equilibrium" had not been studied.

The subject of the etching of platinum metals came under review. Raub said that no special progress had been made in the alternating current electrolytic technique since the publication of the original paper by Dr. Buss and himself.

For platinum-gold alloys containing not more than 80% platinum, the Edelmetalle Institut had used a perchloric acid mixture for both polishing and etching metallographic specimens. The mixture was a fairly conventional one, viz:-

Perchloric acid............	20%
Ethyl ether................	45%
Amyl acetate...............	25%
Water......................	10%

The mixture is dangerous to handle and the temperature should not be allowed to rise above 25°C to 35°C.

Raub indicated that Dr. Spiedel had in hand an investigation on the tungsten-platinum alloy system and had identified an intermetallic compound, though Raub could not recollect in just what percentage of platinum this occurred. A limited amount of work had been carried out on the sintering of metal powders for making specimens required in the Laboratory investigations. Vacuum sintering was preferred, though nitrogen, hydrogen and argon atmospheres were used.

AGFA KAMERA WORKS, TERGERNSTEERLANDESTRASSE, MUNICH.

DATE VISITED

November 26th, 1945.

PERSONS INTERVIEWED

Herr Zeigler - Director - part of time
An assistant to the above

By virtue of their experience in the manufacture and use of precision instruments, Agfa - as a branch of I.G. - had been making the spinnerets for this organization for use by the dyestuffs section, etc. It was explained that Herr Keltemback who was originally in charge of the spinneret section had left Agfa.

Whilst some of the plant for forming and drilling spinnerets had been dismantled, single units of drilling equipment were made available for inspection. During the war period, Agfa had been concerned exclusively with the manufacture of tantalum spinnerets and it seemed almost certain that the quality of the finished articles were very much inferior to United Kingdom production - where far more precautions and much more care were taken to ensure a precision job.

For tantalum spinnerets, the hole making technique was briefly as follows:-

After pressing into the usual "top hat" form, the spinneret was punched with holes on a special laying-out platform. The holing operation was completed by hand drilling with a cylindrical drill which gave a finished cylindrical hole. During this drillin operation the spinneret cup was supported on an "air cushion". Formerly, after the punching operation, a tapered hole was first drilled in the spinneret and finishing was with a cylindrical drill. The progress of the punching and drilling operations was followed fro a binocular microscope.

Finally, the spinnerets were subjected to a heat treatment at a temperature of 400°C to harden them. The mechanism of this hardening of a pure metal by heat treatment was explained on another occasion by Dr. K. Ruthardt of W.C. Heraeus. After fabrication, the spinneret might have a D.P.N. of 80 to 100. After heat treatme in air or carbon dioxide for 10 to 15 minutes at a temperature of 45 the hardness figure rose to 140. It was thought that during the he treatment process a layer of oxide (probably TaO_2) was formed on the spinneret surface and adsorbed, particularly into the outer layers, the metal. A nitriding of the tantalum could also be brought about heat treatment in a low partial pressure of nitrogen at a high temperature. The corrosion resistance of the metal was however impa