ITEM No. 7.

# TELEFUNKEN GESELLSCHAFT FÜR DRAHTLOSE TELEGRAPHIE m.b.h., BERLIN; SPECIAL MATERIALS FOR RADIO VALVES.

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BRITISH INTELLIGENCE OBJECTIVES
SUB COMMITTEE

### TELEFUNKEN GESELLSCHAFT FUR DRAHTLOSE TELEGRAPHIE m.b.h., BERLIN; SPECIAL MATERIALS FOR RADIO-VALVES.

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# Report on Telefunken Gesellschaft für drahtlose Telegraphie m. b. H: Berlin

Special materials for radio valves.

Investigator: E.R. Owen, Asst./Controller, Aircraft Branch, Control Commission for Germany (B.E.)

- Object of Investigation: To obtain information on

  (i) The fabrication of Thorium metal and
  its application in radio valve
  manufacture.
  - (ii) The use of zirconium in radio valves.
  - (iii) Other special metals used in radio valves.
    - (iv) Special getters.

### Summary

The Telefunken organization made from 30-40 per cent. of the total production of radio valves in Germany and occupied countries during the war. Including the occupied countries Telefunken had eight valve factories, the one in Berlin having the largest production. At the Berlin factory the Company developed the application in radio valves of certain special metals, particularly thorium, zirconium and aluminium - plated iron for electrodes, getters, etc. This report describes the fabrication and methods of application of these special metals.

# General Position

During the war the Telefunken organization made from 30-40 per cent. of the total production of radio valves in Germany and occupied countries. It had eight factories making valves; at Berlin, Erfurt, Neuhaus, Litzmannstadt, Prague, Liegnitz, Reichenbach and Ulm, the one in Berlin having the largest output. The peak output of the organization was in December 1944 when, expressed in terms of Reichsmarks, it amounted to 8 million per month. Telefunken aimed at 10 million R.M. by June 1945. (A rate of 10 R.M. per valve will give approximate figures for the corresponding number of valves.)

The rates of production of valves in terms of R.M. per month at the various Telefunken factories during 1943 and 1944 are shown in Appendix I. In December, 1944, the output of the Berlin factory was approximately as follows:-

•	Valves per	month
Miniatures Ordinary Receivers	160,000	
Metal Envelopes	120,000	
Glass Envelopes	50,000	
Transmitting valves	15,000	
	Total 345,000	

Telefunken started making valves in the factory at 71 Sickingenstrasse, Berlin on 1st July, 1939. Until that date the premises were occupied by Osram Werk of the Osram G. m. b. H., K.G. who made lamps there until 1937 and valves thereafter until 1939. Osram moved the lamp-making plant to See Strasse Berlin in 1937.

During the war Telefunken made radio valves of all kinds at Sickingenstrasse, including receivers, transmitting types and miniatures; also components parts and materials such as thorium sheet, wire, getters, leads, etc. They had also one or two smaller plants in Berlin making materials and components.

# Telefunken Gesellschaft für drahtlose Telegraphie m.b. H. Berlin

Location The present Telefunken factory is at Sickingenstrasse 71, Charlottenburg, in buildings that were formerly occupied by the Osram Werk of the Osram G. m. b. H., KG. group. It is in the British sector of the city and within easy distance of the H.Q. of Military Government and the Control Commission.

Map. Reference: Berlin Sheet 5 (Central) F.9.

NOTE: In the Key on the back of the Berlin maps the premises at Sickingenstrasse 71 are still shown as occupied by Osram Werk under the reference No. 324.

Visits: On October 8th, 9th and 15th, 1945.

# Personnel Interviewed

Dr. Erich Weigand - Director and Chief Engineer
Dr. Weth - Director and Head of Valve
Department.
Dr. Statz - Head of Experimental Dept.
Dr. Zuckermann - Assistant to Chief Engineer.
Mr. Stange - Metallurgist.

All the aforesaid persons were very co-operative and appeared to be reliable. Dr. Statz was principally concerned with development of materials for valves and was prepared to go to any trouble to provide information.

# Condition of factory.

whilst the administrative departments had suffered very extensive damage by air attack, the bulk of the premises where valve production was carried on, was almost completely intact. The plant in these premises, however, had nearly all been taken away by the Russians. In large shops where there had been dozens of valve making machines, there were only one or two valve stemming machines, one or two machines for making glass-to-metal seals, and some small machine tools. The firm's expert glass makers were employed in making small glass were such as coctail or liqueur glasses of simple design. Some attempt was also being made to utilize stocks of scrap metal, such as tin plate, to make simple domestic utensils such as tea strainers.

Effort was being made to restart the manufacture of radio valves in a small way. In October they were making the simple German types R.E. 164 and R.G.N. 354 for civilian sets; and in November they hoped to make a start on types D.K.E. VY2 and D.K.E. V.C.L.11

# Metals used in Radio Valves

The principal metals used by Telefunken in the manufacture of radio valves are shown in the schedule in Appendix 2 which gives also the purposes for which the metals were applied and the suppliers of the materials. Most of the metals listed are well known to radio valve makers everywhere, and in the limited time available for the present investigation attention was devoted almost exclusively to special, less generally used, materials chiefly thorium, zirconium, one or two alloys and special getters.

# Thorium

Telefunken bought Thorium metal powder from Degussa (Deutsche Gold und Silber Scheideanstalt) Frankfurt/Main and Berlin. This powder contained at least 97% Thorium metal and usually 98-99.5% total Thorium. The chief impurities were oxygen (as Thoria) calcium, iron and sodium. Iron was generally about 0.1%; sometimes it exceeded this amount. If iron exceeded 1 per cent. the Thorium was used only for making getters, and no attempt was made to work it into rod or sheet. Calcium oxide was less desirable than iron and for consistently good results in working the metal, the calcium content should not exceed 0.05%.

Degussa produced this Thorium powder by direct reduction of thorium oxide (thoria) with calcium chippings and calcium chloride. A mixture of thoria (obtained from Auer Gesellschaft A.G. Berlin) calcium and calcium chloride in the proportions.

Thoria 100 Kg.
Calcium chippings (Theoretical weight + 10%)
Calcium chloride 25 Kg.

is rammed into a pot made of thin iron sheet and heated in a gas furnace to 1100°C in vacuo. The reaction takes about 15-30 minutes and when complete argon is introduced into the pot and the contents are allowed to cool in this inert atmosphere. The contents are then emptied, crushed, milled, digested with water while stirring, and with hydrochloric acid. The metal powder is separated out on a shaking table and dried. The production of Thorium powder by Degussa was investigated thoroughly by a B.I.O.S. Team in August, 1945.

All the Thorium powder produced by Degussa during the war was supplied to Telefunken Berlin for use in radio valves. Degussa produced the metal by a batch process and their cutput depended chiefly on the orders placed by Telefunken. The quantities absorbed by the latter varied from about 150 Kg. to a maximum of 250 Kg. per month. According to information furnished by Degussa their total cutput of Thorium powder during the war was about 5 tons.

Telefunken used the powder for working into sheets and rod and, in powder form for coating valve parts and as a constituent in getters.

# L. Thorium Sheet

Before pressing the powder was passed through a sieve with meshes .057 x .066 m.m. No preliminary washing treatment was applied. For rolling into sheet about 550 grams of powder was pressed into bars 150 x 25 x 25 m.m. using a steel mould and an hydraulic press as commonly used for pressing tungsten or molybdenum powder. The applied pressure was about 3000 Kg/sq. cm.

# Sintering

The pressed bars were first dried in a vacuum tubular furnace at about 60°C and then sintered in vacuo at 1600-1630°K (1327-1357°C) in either a high frequency or resistance furnace. In the high frequency furnace three dried bars rested on pieces of tantalum or molybdenum in a carbon tube inside a glass container like a large thermos flask with a connection to a vacuum pump to evacuate the flask. In the resistance furnace method, which was preferred,

the furnace comprised a horizontal porcelain tube (Pythagoras porcelain) closed at one end placed inside a wider procelain tube open at both ends and wound with an element of platinum ribbon. The outer tube was mounted in an outer steel casing the space between tube and casing being packed with a suitable refractory such as magnesia. The Thorium bars, up to three in number were charged into the inner porcelain tube and the open end of the tube which projected well beyond the end of the outer porcelain rube was closed with an airtight stopper. The stopper carried an outlet tube which was connected to a vacuum pump.

The bars were sintered for 3 hours in vacuo; they were not presintered between the drying and the sintering.

# Rolling:

After rounding the edges the sintered bars were hammered cold to a thickness of about 18 m.m. and a length of 180 m.m. and cut into two lengths of 90 m.m. each. The 90 m.m. bar was rolled cold across the length (to get the width) and when the starting thickness was reduced by about 25-30 per cent. in about 4 passes, the bar was annealed in vacuo. To reduce the thickness from 18 m.m. to 1 .m. the bar had to be annealed 9 times, and for about 1 hour at 1600°K (1327°C) each time. Beyond 1m.m., sheet was frequently rolled down to .05 m.m., reducing the thickness in stages of 15-20 per cent in 3 or 4 passes and annealing after the early stages. Annealing of sheet during rolling was done by rolling the sheet up and placing the roll of sheet in a crucible furnace which could be evacuated.

Rolling was done cold, in 2 high mills with rolls 200 m.m. diameter and 250-300 m.m. wide followed by finishing mills with rolls the surface of which had a hardness of 90 as tested by the shore scleroscope.

# 2. Thorium rod and wire

Rod and wire were made from either the larger pressed bar 150 x 25 x 25 or a smaller bar 180 x 7 x 7 m.m. (which on sintering was reduced to about 545 m.m. section) according to the final diameter required. The smallest diameter of wire made was 0.5 m.m. and was manufactured from the bar by swaging throughout; rod was not drawn down as in the case of tungsten or molybdenum.

The standard size of rod produced for the fabrication of valve parts was 4.5 m.m. in diameter. Swaging was done cold in rotary machines with intermediate annealing as in the case of rolling. The number of annealings however, depended a great deal on the quality of the starting metal. If the metal was particularly free from embrittling impurities such as iron or lime, one or two anneals would suffice. The reduction of diameter per pass on swaging was

about 8 per cent. down to 4.5 m.m. diameter, and a little higher for diameters below 4.5 m.m.

# Applications of Thorium in Radio Valves

### 1. Powder

Thorium powder was used for coating anodes and other parts of valves and as a constituent of special types of getter. It was claimed that Thorium had a high gettering power and when parts coated with thorium were introduced into a valve, a higher vacuum inside the valve was possible.

# Process for the coating of valve parts with thorium

The process employed was the cataphoretic deposition of thorium (i.e. deposition from a paste or solution containing thorium in a colloidal state by means of an electric current). This process will be described under the following heads:-

- (i) Proparation of working material.
- (ii) Preliminary treatment
- (iii) Deposition of thorium.
  - (v) Sintering.

# (i) Preparation of working material

The material used was a paste made from thorium powder, pure methanol, acetic acid, nitric acid and aluminium nitrate of the following specifications:-

Thorium powder - Iron less than 0.5%

Calcium less than 0.1%

Methanol - pure (supplied by Schering A/G)

Acetic acid - 10% solution

Nitric acid - 1% solution.

Aluminium nitrate Al (N03)3.9 H<sub>2</sub>O (pure) - 7.5 grs. dissolved in 100 c.c. distilled water.

To prepare the paste, thorium powder (after sieving) methanol and acetic acid are charged into a porcelain mill, capacity 5 litres, in the following proportions:-

Thorium 150 grams Methanol 300 c.c. Acetic acid 3 c.c.

The mill contained balls each 20 m.m. in diameter and weighing 1200 grams. The charge is ground for 48 hours and after grinding poured through a large glass funnel into a 3 litre flask,

2100 c.c. of methanol is poured into the mill to wash out the remainder of the paste into the 3 litre flask, into which a further 15 c.c. of dilute acetic acid is added. The contents of the flask are well shaken. Before use the contents of the flask are shaken thoroughly and transferred in three equal portions into 3 flasks, each 1 litre capacity, taking care that no hard paste sediment is left behind. The paste as it is filled into each 1 litre flask is shaken vigorously. Each of the portions in the 3 flasks then receives an addition of 10 c.c. of the aluminium nitrate solution and 10 c.c. of dilute nitric acid (1%). The nitric acid is necessary to prevent the formation of dimethylamine, (CH<sub>3</sub>) NH or Trimethylamine (CH<sub>3</sub>) N which would cause the thorium paste to coagulate.

The above quantities of starting materials should yield 2500 c.c. of paste. When sieving the thorium powder the operator should wear goggles to protect the eyes. Also the paste is inflammable and a fire proof hood should be worn.

# (ii) Preliminary treatment of parts.

In order to obtain a uniform coating of thorium, parts of metal e.g. nickel, must first be annealed in a stream of hydrogen to clean the surface. During annealing parts must be so supported as to allow the hydrogen access to all parts of the surface. Soldered parts should be annealed at 800°C, and welded parts at 1000°C, for 10 minutes.

# (iii) Deposition of thorium

Generally the deposit of thorium must not exceed 2.5 mg. per sq. cm. as thicker layers tend to peel off on sintering. An exception to this rule was anodes made of very thin sheet metal e.g. the anode of valve type RV12P2000 which comprised two half circles made of thin iron coated with aluminium. The Thorium deposit on these thin pieces tends to be displaced relative to the underlying metal and a thinner coating is therefore necessary. The maximum thickness of the deposit in such cases is 1.3 mg. per sq. cm. of surface.

Thorium was deposited on parts from the paste or solution described above either on a Getter Automatic Machine or a separate cutaphoresis apparatus (particulars of which were not available at the time of the visit) the former being advantageous for the treatment of parts with a simple profile as they allowed a higher rate of handling. The anodes made of iron or mickel were as nearly as possible of the same profile as the parts to be treated which formed the cathodes in the bath. No hard and fast rule obtained as regards electrode spacing; spots with particularly short electrode spacing should be avoided as the Thorium is loosely held at such spots and peels off on sintering. In most

types of parts a satisfactory deposit of thorium depends on the development and use of the right anode.

Direct current from a 200 volt supply was applied. using a potential divider, resistance 850 ohms and carrying 1.5 amps. The potential divider is protected against overloading by an automatic cut out and the cataphoretic apparatus had a device for detecting short circuits. A test for short circuits was made before each deposition. deposition potentials varied between 15 and 60 volts and the time of "deposition between 5 and 20 seconds depending on the thorium content of the paste used. As thorium supplies became scarcer, pastes poorer in thorium had to be used and deposition potentials and time of deposition had to be increased with these pastes to give the standard thorium deposit. The potential, however, had to be carefully controlled; it could not be increased at will owing to the evolution of gas which in the case of parts with an intricate profile tended to collect in corners and recesses and prevent the adhesion of thorium. The potentials and time to deposit for various pastes are, however, easily determined. The deposition of thorium on welded edges or rims of valve anodes can be prevented by covering up the edges or rims. If this is not feasible the thorium can be scraped off after sintering. For some valve types with large electrode currents, essential to keep the inner surfaces entirely free from thorium, and in such cases traces of thorium on the inner surfaces were removed by means of a revolving tungsten-wire brush.

After deposition the parts were subjected to a blast of hot air in order to spread any adhering paste from the bath evenly over the surface and so dry the deposit. Drying had to be done carefully especially in the case of iron parts which rust very readily.

The quantity of thorium deposited was checked by weighing in the case of anodes and by measuring the thickness of the deposit in the case of other parts. It was found that by keeping strictly to the limits stated above (2.5 mg/sq.cm. or 1.3 mg/sq.cm.) for the deposit only a small quantity of thorium was wasted through peeling off the deposit. Care had to be taken during the coating process that the anodes of the bath were continually kept free from adhering thorium. Also in the clamping of the metal parts all contact surfaces had to be kept bright to keep down the electrical resistance.

The pastes used for the cataphoretic bath are inflammable and the presence of conbustible thorium powder mixed with methanol increases the danger of fire. The deposition was carried out in glass containers placed inside a tin box, and

each container was fitted on its upper flange with a circular tube with several holes in the circumference and connected to a carbon dioxide bomb. By means of this tube the bath could, in case of fire, be immediately enveloped in a screen of carbon dioxide. In cleaning the bath anodes also care had to be taken that large quantities of loose dry thorium scrap did not accumulate as thorium powder readily ignites merely by friction. The methanol vapours from the bath were drawn off by efficient ventilation and thorium scrap was not allowed to collect in the fume offtake where it could also start a fire.

# (iv) Sintering.

After deposition all parts were sintered in a vacuum furnace; nickel or iron parts were subjected to a temperature of 800-1000°C. and carbon parts 1500-1600°C.

Thorium coating was applied to anodes and screens of power valves, e.g. type R.V.271, smaller valves e.g. type R.V.12P2000 and transmitting valves. It was particularly suitable for anodes made from iron, nickel, molybdenum and carbon. Thorium is a good getter and when used as a coating on anodes helps to produce and maintain a higher vacuum in the valve. Samples of thorium-coated parts were obtained.

Thorium powder was also used in the manufacture of special getters which are described under "Getters" belov,

# 2. Thorium Rod and Sheet.

It was stated that thorium was used only in short wave valves. Thorium rod machines very well like free-cutting brass and parts with complicated profile can be produced accurately from rod or bar. Thorium sheet was used extensively in magnetrons.

# Thoriated Tungsten Wire.

Telefunken also made their own thoriated tungsten from pure tunstic oxide bought from Osram and thoria from Auer Gesellschaft Berlin. They found that 1.8% Thoria gave the best results in wire for impulse tubes. Thoriated wire was used for cathodes and springs particularly transmitting types such as L.S.180.

The thoriated wire was made at a small plant put up during the war at the Brock Brauerei Schwiebussa Strasse (opposite Tempelhof Aerodrome) now in the Russian sector. The mixed

tungstic oxide and thoria were pressed and centred into bars  $20 \times .5 \times .5 \text{ cm.}$ , swaged and drawn in the usual way. The output was about .5 kg. per day of wire.

# Zirconium.

Zirconium was applied to the surface of anodes screens and grids in transmitting valves particularly when these parts were made of nickel, iron or carbon. The starting material was Zirconium hydride which was made into a paste and the latter was applied to the parts either by spraying or cataphoretic deposition.

# (i) Deposition.

The paste was made up from the following ingredients:Zirconium hydride (at least 80% Zr H4)
Methanol (pure)
Aluminium Nitrate (Al(NO<sub>3</sub>)3: 9H<sub>2</sub>O
free from Sulphur and Alkali

To make the paste 150 grams of zirconium hydride and 300 cc. methanol were introduced into a porcelain mill, 5 litre capacity, containing 1300 gram balls. The charge was ground for 24 hours. The mill rotating at 70-80 revolutions per minute. After grinding 2100 cc. methanol and 45 cc. of aluminium nitrate solution (7.5 grams per 100 cc. of water) were added. This yielded about 2400 cc. of paste.

The zirconium was deposited on the valve parts in the same way as described for Thorium.

# (ii) Spraying.

The paste used for spraying was made from:Zirconium Hydride (at least 80% Zr H<sub>4</sub>)
Xylene
Paraffin (Solid - M.P. 42-40 0)
Naphthalene (pure)

Methanol (pure)

A paraffin - naphthalene solution was first prepared by dissolving 25 grams paraffin and 50 grams naphthalene in 2000 cc. Kylene and adding to this solution 100 cc. methanol. 400 cc. of this solution was then mixed with 200 grams zirconium hydride and the mixture was ground for 10 hours in a porcelain mill, 1 litre capacity and containing 175 grams

porcelain balls, revolving at 72 r.p.m. This mixture yielded 400 cc. paste. To test the paste two lots of 400 cc. from the mill were mixed together in a litre powder bottle (widemouthed) and a small sample of the paste was sprayed on two anodes of any straight forward type in production at the time.

### Sintering.

After deposition or spraying the parts were sintered in vacuo at temperatures of 800-1000°C. in the case of iron or nickel parts and 1500-1600°C. in the case of carbon.

NOTE: Valves fitted with zirconium coated anodes should not have a deposit (mirror) of magnesium inside.

### Iron.

Owing to shortage of money and anode materials such as nickel, Telefunken used a great deal of specially prepared iron wire sheet and strip for anodes of most types of valves.

"E" Iron. This was a very pure iron containing less than .04% of carbon, well "killed" with a trace of aluminium left behind in the metal. Sheet and strip were supplied by Thyssen and wire by V.D.M. Altena. Krupps also supplied a pure iron known as W W-iron, E- and W W-iron were used for anodes, screens, leads and supports; the iron was sometimes pure, sometimes plated with nickel or coated with special materials such as thorium.

"P2" - Iron. The most extensively used composition was iron plated on both sides with aluminium, the plating being from 10 to a maximum of 15 microns thick. When this material is heated in vacuo to 680°C. the aluminium and iron alloy and the surface turns black. In this condition it presents a surface with heat radiation properties equal to 85% of the perfect black body. If the material is heated above 900°C. the surface becomes bright again and looses its black body characteristics. Telefunken claimed that below 900°C. this aluminium coated iron is superior to carbonised nickel, and they used for anodes of strong receiver valves, medium transmitting valves and large amplifiers where the anode load is high.

"PN" - Iron. Where the clearance between anode and grid is small an aluminium coated surface cannot be used and in such cases Telefunken used iron plated on one side with aluminium and on the other with pure nickel. Anodes made of "PN" iron had a nickel plated surface on the inside and an aluminium plated surface on the outside.

Both "P2" and "PN" iron were supplied by Trierer Walzwerke who turned out aluminium "plated" or "clad"iron in large quantities for containers for food and other commodities. It transpired that

Telefunken discovered merely by accident that "P2" had useful properties for valve parts.

### Getters.

In addition to the well known types of Getters using barium - magnesium - aluminium alloy combinations Telefunken used two special types known as "Bato" and "Ceto" in which the basis is thorium powder.

# "Bato"Getters

For the "Bato" getter a meixture was first prepared from the following ingredients:-

Aluminium - barium alloy powder	<b>9</b> 72	grams
(50% Al 50% Ba)		
Thorium metal powder	2942	tt
Iron oxide (Fe <sub>2</sub> 0 <sub>3</sub> )	116	18
· 2 J.	4000	11

The aluminium-barium alloy was applied by I.G. Farbenindustrie and was in granules about .07 mm. diameter. The thorium powder came from Degussa through the Auer Gesellschaft Berlin and the iron oxide was prepared from iron oxalate (No.71544) by Schering A.G.

The ingredients were mixed together for two hours in a mixing machine and filled into glass bottles with ground stoppers. The mixture was used in lots as required and pressed into tablets which were assembled on flags of nickel in an automatic getter machine in the usual way.

"Bato" getters require a slightly higher temperature than barium-magnesium getters to flash; they were therefore used chiefly in valves with hard glass envelopes for example medium transmitting valves.

# "Ceto" Powder.

The "Ceto" getter was used in the form of a paste which was applied to the surface of metal components, usually anodes.

"Ceto" powder was made from a mixture of thorium metal powder and an alloy of cerium-misch metal and aluminium, the thorium forming 80% of the mixture.

# (i) Preparation of misch-metal-aluminium alloy.

Composition: Misch-metal 72% Aluminium (99% pure) 28%

The cerium-misch metal which was supplied in the form of blocks under paraffin oil was freed from oil by washing in xylene or petrol and brushing with a steel wire brush until it showed a metallic lustre. The misch metal blocks and aluminium blocks were charged in the ratio of 2.6 parts to 1 part by weight respectively, in alternate layers into a hard porcelain crucible (or if not available an iron crucible) the quantity charged depending on the circumstances. The porcelain crucible was placed inside a large iron crucible about 50 mm. longer (owing to danger of spurting of the charge). The crucibles were placed inside a vertical vacuum furnace consisting of hard porcelain or iron tube closed at one end and wound with platinum ribbon. The tube was connected through a metal ground top to pumping equipment and the top had a window through which the melt of the crucible could be observed. After evacuating the furnace the temperature was raised to the reaction temperature of the mixture normally about 750°C. The reaction is indicated by an agitation in the melt, a sudden rise of the temperature to about 1.500°C. and spasmodic evolution of gas. When the reaction subsides the current can be switched off at once but the pumping must continue for 10 minutes further or longer. The glass-hard regules was allowed to cool in vacuo and then transferred into a porcelain or higher mortar for grinding to the fineness of gravel.

# (ii) Pressing.

600 grams of the misch metal alloy and 2400 grams of thorium powder were mixed in a mixing machine for 30 minutes and pressed in an hydraulic press under about 2 tons sq/cm. into rods 12 x 12 mm. section. The length of the rods was determined by the dimensions of the hot zone of the furnace used to sinter them.

# (iii) Sintering.

The sintering of the pressed bars was done in a vertical or horizontal furnace. In either type the tube was used closed at one end and wound with resistance wire or tape and having at the other an airtight closing device connected to a vacuum pump. In the vertical furnace a tall crucible was used and in the horizontal furnace boats were used, to contain the pressed bars. Crucibles, bolts and tubes had to be made of hard porcelain or quarts. Furnace tubes of quarts particularly in the horizontal furnace were spoilt if they came in contact with any fused"ceto" alloy. Fusion of the pressed bars had to be prevented by exact control of the temperature not only to preserve the equipment but also because fused alloy is ductile and therefore difficult to powder. More over the alloy in the sintered condition has better gettering qualities.

Before starting to heat, the pressure in the furnace was reduced to a point not higher than 0.001. The current input was controlled in such a way that with the pump running continuously, the temperature of the charge reached 1000 C. after about 3 hours. The temperature was held at this level for 2 hours after which the

current was switched off. Fifteen minutes later the pump was stopped and the charge was allowed to cool in a good vacuum for at least 4 to 5 hours and preferably overnight.

# (iv) "Ceto" Powder.

The rods tend to stick together during sintering and had to be parted carefully from each other and brushed with a steel-wire brush. They were reduced to powder first by hand in a porcelain mortar, taking not more than 50 grams "Sinterlings" at a time and reducing them to a coarse powder which was sifted through a silk fibre sieve No.25 (meshes 0.02 mm. x .02 mm.) This sifting produced powder ready for use. The coarser material on the sieve was moistened with methanol and ground in hard porcelain ball mill in an atmosphere of nitrogen for 8 hours. The ground product must be removed from the mill dried and sieved again with the greatest care because of the great risk of it igniting.

# (v) Storage.

"Ceto" powder is inflammable and ignites readily with friction or impact; sometimes there is a tendency to self-ignition particularly if it contains much nitride or moisture absorbed from the atmosphere. It must therefore be stored in a cool, dry fire-proof container and protected from risk of friction or impact (e.g. bottles with ground glass stoppers are unsuitable). The quantity of "Ceto" powder per container should not exceed 3 kg.

# (vi) Precautions.

Because of the risk of ignition the eyes should be protected when handling the powder; it should not be handled near open fires or furnaces and supplies of sand for fire-extinguishing should be readily available at all places where it is handled.

Scraps containing the powder must not be placed in dust-bins; they should be disposed of safely elsewhere.

"Ceto" should be delivered or shipped only as sintered rod.

# (vii) Application.

As already stated "Ceto" was used as a coating getter. A paste was prepared consisting of 5 parts by weight of "Ceto" powder and 1 part by weight of a 2.5 per cent collection solution. The paste was applied to metal parts with soft hair brushes and the parts were then sintered in vacuum, iron or nickel parts at 1100°C. and silver at 800°C.

# Conclusions and Recommendations.

- 1. In connection with the metals used during the war for the production of radio valves at the Telefunken works, Berlin, whilst there is now nothing left of the plant used for the manufacture and application of these materials yet a great deal of useful information about manufacture and application can be obtained from the firm's technical and administrative personnel who are still available for interrogation in the British sector of Berlin.
- 2. During the visits reported here the chief items of novelty, at least from the point of view of British radiovalve technique, that emerged, were thorium, zirconium and aluminium plated iron. During the war zirconium coated parts were used in U.S.A. valve practice but not by British valve makers; and as far as is known neither country used thorium or aluminium plated iron parts. As compared with the expensive carbonised nickel used largely by British and American valve makers, aluminium plated iron would be considerably cheaper. (It is understood that the material is made by rolling iron between aluminium sheets in a manner similar to the rolling of "Alclad").
- 3. Telefunken was the only firm in Germany (and as far as is known, in the world) that made thorium powder into sheets, rod, and wire. In view of the advantages claimed for this metal over molybdenum (namely that it is "cleaner" and helps to maintain a vacuum better in the valve) further investigation of the application of the metal to radiovalve production by manufacturers might be worth while.

Appendix 2

Principal metals used in Radio Valves

Metal	Composition and Form	Uses .	Supplier of Metal
"E" Iron	Pure ingot iron; wire, tape and sheet.	Supports, Leads Anodes, Screens	Wire: V.D.M. Altena, Tape and Sheet: Thyssen
"EA" Iron	Selected "E" Iron with superior "Vacuum" properties; .05% Al. (metallic) and .05% C. deep drawn sheet and tape.	Anodes	Thyssen
"P2" Iron	Special iron plated on both sides with aluminium alloy . Sheet	Anodes	Trierer Walzwerk
"FN" Iron	Special iron, plated on one side with aluminium alloy and on the other with Nickel. Sheet	Anodes	Trierer Walzwerk
Nickel- plated Iron	Pure ingot iron wire plated with nickel. Ni-plate 10-15% of diameter.	Grid wire	V.D.N. Schwerte
Chrome-Iron	75% Iron, 25% Chromium allo <b>y</b> wire.	Pin-leads through soft glass.	Heraeus Vacuum- schmelze.
Nickel-Iron	54% Iron, 46% Nickel wire and tape	Leads to cathodes and heaters where low thermal conductivity is involved.	Krupp A.G.

Metal	Composition and Form	Uses	Supplier of Metal
"Fink" Wire	Nickel-iron core, clad with copper (Copper-ciad wire)	Lead wire for soft glass	Osram, Berlin
"E" Nickel	99% Nickel containing magnesium. Free from volatile metals e.g. zinc, cadmium, lead. Tested for impurities during melting. Wire strip and sheet.	Supports, leads, anodes, etc.	V.D.N. Schwerte
Cathode- Nickel "A"	99.8% Nickel with .025065% Mg. Tubing	Cathode tubes for indirectly-heated cathodes	V.D.N. Schwerte
Cathode- Nickel "C"	99.8% Nickel with .06515% Mg. Tubing.	Cathode tubes for indirectly-heated cathodes with specially good emission.	Krupp A.G. or Schoeller, Hellental (Eifel)
Manganese- Nickel	"E" Nickel containing l-2% Manganese Wire	Supports with higher mechanical strength.	V.D.N. Schwerte
Copper	Electrolytic oxygen-free wire and strip. Commercial grades low in oxide and other impurities e.g. zinc	For parts inside the valve.  For parts outside the envelope.	Siemens- Metallwerk
Manganese- Copper	Copper containing O.5-1% Manganese Vacuum - melted.	Bases and sockets	Heraeus Vacuum Schmelze
Nickel- Copper	55% Copper, 45% nickel, wire strip and sheet (Not identical with Constantan; must be free from impurities such as manganese, zinc, etc.	Leads and various parts. Non-magnetic parts for television tubes	V.D.N. Schwerte

Metal	Composition and form	Uses	Supplier of metal
Rolled Bronze	94% Copper, 6% Tin, wire, strip and sheet.	Non-magnetic parts in valves and television tubes.	Sundwigen Metallwerke.
Nickel- clad copper	Electrolytic copper wire low in oxide content, clad with nickel free from zinc. Copper wire plated with nickel to a thickness of 10% of diameter.	Grid side rods	D.E.W.
B.207	Iron with 18% chromium, 1% Molybdenum. Wire and strip	Leads and pins for sealing to glass, and caps for metal valves.	Harekort Eicken
Kovar	53% Iron, 27% Nickel, 19% Cobalt, 0.4% Manganese	Leads and pins for sealing to glass.	Heraeus Vacuumschmelze
Tungsten	Pure tungsten wire and rod	Leads and supports. Ground and etched for sealing-in.	Osram.
G.7.	Thoriated tungsten (.75% Thoria) wire	Filaments for directly heated cathodes, (emission or paste)	Osram.
Thoriated Tungsten	Tungsten containing 1.2-1.8% Thoria Wire	Filaments for directly heated cathodes with Thorium emission.	Telefunken
B.S.D. Tungsten	Tungsten containing traces of aluminium silicate and al. oxide. Wire	Heaters in indirectly heated cathodes and cathodes with pure tungsten emission.	Osram.
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Metal.	Composition and Form	Uses	Supplier of Metal
N.S. Tungsten	Tungsten with traces of aluminium silicate. Wire.	Heaters in indirectly heated cathodes and cathodes with pure Tungsten emission.	Osram
Molybdenum	Pure molybdenum wire, rod, and sheet. Plain or nickel-plated.	Supports, side rods, for grids; grid-winding. Anodes for transmitting valves working at high temperatures.	Osram or D.E.W.
Tantalum	Pure tantalum wire strip and sheet.	Grids. Anodes and cathodes in transmitting valves. Special supports.	Siemens and Halske
Niobium	Pure niobium strip and sheet.	Anodes, cathodes.	Siemens and Halske.
Thorium motal	97% Thorium with remainder mainly oxygen. Sheet, rod, wire, powder.	Electrodes in magnetrons. Parts of short wave valves. Powder for coating carbon and metal anodes. Getters.	Degussa, Frankfurt Main (Powder) Telefunken (wire, rod and sheet)
Zirconium	Pure zirconium powder reduced from zirconium hydride paste.	Coating on anodes and grids.	Degussa (powder).

<u>+</u>.

# APPENDIX . I.







