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I.G. FARBENINDUSTRIE—OPPAU WORKS LUDWIGSHAFEN

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE

LONDON — H.M. STATIONERY OFFICE

I.G. FARBENINDUSTRIE - OPPAU WORKS
LUDWIGSHAFEN

Reported By

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Min. of Supply

CIOS Target Number 21/22 (a)
Metallurgy

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

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COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE.

SECTION 21 - METALLURGY.

REPORT ON EXAMINATION OF BLACK LIST TARGET 21/22(a).

Reports have been received in this country that the I.G. Farbenindustrie had turned over the whole, or a part, of the plant originally built for the manufacture of Carbonyl Nickel to the manufacture of Carbonyl Iron Powder. The object of the visit of the Team was to establish the facts.

It was found that the Carbonyl Nickel Plant had been maintained in operation and produced an average of about 350 tons of Nickel per month up to December 16th, 1944.

An entirely new installation had been erected for the manufacture of Carbonyl Iron Powder. This plant was also in operation up to December 16th, 1944, with an average production of 100 to 110 tons of Iron Powder or other product, per month.

Further Nickel Target.

It was ascertained that a new plant is under construction for the production of electrolytic nickel by the "Frose" process at Aschersleben and is in a condition practically ready to commence operation. This plant, embodying the latest developments, should be worthy of examination. It is stated to have a capacity of 20 to 30 tons per month.

Documents.

During the period from December 16th, 1944, to the date of visit, all documents of importance and practically all the chief personnel of the operating and research staffs, had been evacuated to Heidelberg. As a result, no detailed precise information on tonnage production or details of development or operation, were available. The general data outlined in this report were obtained by interrogation.

On following up the target to the town of Heidelberg, April 18th to 24th, Dr. Leo Schlecht was located and interrogated. It was found that some of the documents relating to Metal Powder had been hidden in a small village about five miles south of Ludwigshafen. These were recovered and a further report, if necessary, will be presented on receipt of the documents in this country.

Principle of Manufacture.

The manufacture of Carbonyl Iron or Nickel at the Oppau Works, is based on the well-known principle that both of these metals under suitable conditions of temperature and pressure combine with CO gas to form a Carbonyl and by variation of the temperature and/or pressure, these Carbonyls decompose with the liberation of CO gas, which is recirculated, and the deposition of the metal. The principles underlying this process were worked out by Mond in this country and formed the basis for the manufacture of Nickel by the Mond Nickel Company. The same principles have been adopted in recent installations for the production of Carbonyl Iron Powder in U.K. and U.S.A.

The Target examined was the Works of the I.G. Farbenindustrie at Ludwigshafen, with particular attention to the section at Oppau engaged in the manufacture of Nickel and Iron Powder.

The buildings occupied by these manufacturing processes are marked as follows on the Works Plan:-

Nickel Plant	46, 53, 217, 286, 287.
Iron Powder Plant	309, 310, 311.
Sintering of Iron Powder .	510.
Raw Materials Storage ...	706.

Condition of Plant.

The Works have been subjected from time to time to heavy air attacks and also suffered from artillery fire during the Occupation of the town. Certain sections of the Works have been completely demolished and others subjected to varying degrees of damage. This aspect will, however, be more fully dealt with in the Reports of the other Teams visiting the Target and reporting on the various sections.

The condition of the above-mentioned buildings may be summarised as follows:-

46 & 53:	In good condition and easily repaired.
217:	Totally destroyed.
286 & 287:	Intact, - no damage.
309 & 311:	In fairly good condition and easily repaired. All equipment seemed to be in operable condition.
310:	Completely destroyed.
510:	This building was the Central Research Laboratory. It is badly damaged on the upper floors by fire, but can be easily repaired. Lower floors in fair condition.
706:	Building badly damaged. Only equipment, a crusher for nickel matte, can be easily repaired.

Operations.

This plant operated fairly normally until Septembe 1944, and then slowed down to about 70 per cent capacity by December. All production ceased on December 16th, 1944, following a heavy air-raid during the night of December 15th 16th, which destroyed the mains which supply the gas to the plant and cut off electric power supplies. Subject to the repair of these supply mains, on which both the Nickel and Iron manufacture are dependent, manufacturing operations could be resumed within a short period, say, three months.

The following information was obtained by interrogation of Dr. Klippel, the head of the Nickel Department, who was interviewed at the Oppau Works, and from Dr. Leo Schlecht, the Head of the Metal Powder Department of I.G. Farben, who was interviewed in Heidelberg on April 23rd, 1945.

MANUFACTURE OF NICKEL.

Raw Material.

The principal raw material used at Oppau is Nickel Matte normally containing 40 to 45 per cent Nickel, 45 to 50 per cent Copper, and 8 to 10 per cent Sulphur. This was imported pre-war from Canada and at the outbreak of war the stocks held were equivalent to about two-and-a-half years' supply. During the war, supplies have been received from France and Norway, and during 1943 and 1944 between 6,000 and 8,000 tons from Petsamo. A secondary material, Rohstein, containing 8 to 12 per cent Nickel, was also imported from Petsamo. This material contained 4 to 6 per cent of Copper with the remainder Iron, Sulphur and small quantities of precious metals.

Process.

1. The Nickel Matte was unloaded into storage, crushed as required to a maximum of 10 m.m diameter in Building 706, and transferred by wagon to the Nickel House.

2. The Crushed Matte was charged into high-pressure reaction chambers. These chambers are cylindrical in shape, about 8 metres high, 500 m.m. diameter, with apertures 120 m.m. diameter, top and bottom. They are constructed of creep-resisting steel, containing about 0.5 per cent each of Chromium, Tungsten and Molybdenum, and the inner surface is corrugated.

These reaction chambers are constructed and operated in groups of four, with a combined capacity of 21 to 23 tons of Matte per group.

The chambers are heated to a temperature of 250 to 260°C and CO gas preheated by superheated steam and electric heaters passed through at a pressure of 200 Atmos. for about four days. Under these conditions, the CO gas combines with about 85 per cent of the Nickel (and some iron), to form Nickel (and iron) Carbonyl $Ni(CO)_4$ which is volatile, leaving the impurities as a residue in the chambers.

The issuing gases are passed through copper gauze and flannel filters to extract dust and are then cooled to condense the Carbonyl - Nickel Carbonyl has a boiling point of 43°C, - the liquid Carbonyl being collected by traps and the CO gas recirculated. The liquid Nickel Carbonyl passes through a pressure-reducing trap and is collected in tanks.

The residue from the reaction chambers has been despatched during the war to the Norddeutsche Affinerie, Hamburg, for the extraction of copper and the precious metals. This residue contained about 78 per cent Cu & Ni, 20 to 25 per cent Sulphur and its content of Precious Metals was stated to average -

	Per ton of Residue.
Silver	150 Grams.
Gold	2 to 3 "
Platinum	5 to 6 "
Palladium	5 to 6 "
Other Rare Metals ...	0.5 "

3. The Nickel Carbonyl is collected in tanks or vessels under an atmosphere of CO, 2 to 5 Atmos. pressure, to prevent volatilisation and to prepare Nickel metal of high purity, the crude Carbonyl is subjected to redistillation to separate the Nickel Carbonyl from other Carbonyls - mainly Iron.

$Ni(CO)_4$ has a B.Pt. of 43°C as compared with $Fe(CO)_5$ - B.Pt. 103°C, and the fractionation is conducted under a pressure of 0.4 to 0.5 atmos. with a maximum temperature of 70 or 80°C. This yields a Nickel Carbonyl of high purity.

4. The pure Nickel Carbonyl is then passed to the decomposition process which is carried out in eleven chambers or cylinders. The liquid Carbonyl passes slowly from a reservoir into a vapourising cylinder where it is heated by steam coils within the liquid and the vapour then passes into a cylindrical chamber at a temperature of 220 to 240°C, under a pressure of 200 to 400 m.m. w.g. over atmospheric. The $Ni(CO)_4$ decomposes into Nickel and liberates CO gas which is recirculated.

The character of the powder is dependent upon the speed of operation. With a slow rate of production of 300 to 400 Kgs. per week, the powder is of a very heavy type and is extracted by a Beth filter bag system. The normal light Nickel powder is produced with an operating rate of

1300 to 1500 Kgs. per day per unit. The maximum rate of operation was given as 1700 Kgs. per day but under such conditions the operation is dangerous and the Nickel is deposited in the form of light filaments or wool which cannot be extracted.

With eleven decomposition chambers in operation, the production was given as about 350 tons of Nickel per month with a record production of 425 tons.

5. The Nickel Powder is crushed in a Ball Mill, screened, and barrelled for despatch.

6. A small portion of the Nickel Powder is sintered by heating in an electrically heated furnace for twenty-four hours at 1000°C in an atmosphere of hydrogen. This sintered material was despatched mainly to Heraeus and to Universities for experimental purposes.

7. The main bulk of the Nickel Powder made was despatched to Krupps, and during the war to D.E.W., Krefeld.

Stock.

It was stated that the stock of Nickel Matte and material in progress in the Oppau Works was about 100 tons Ni. The main stockyard for Nickel Matte is at Schriesheim, near Heidelberg. The stock in this yard was stated to be 600 to 800 tons of Matte.

Recovery of Nickel from Residue.

It has been reported that steps have been taken, during the war, to conserve Nickel by treatment of the residue from the reaction chambers. In the normal operation, about 85 per cent of the Nickel in the matte is converted into Carbonyl in about one-hundred hours. The reactions to extract the remaining Nickel from the residue proceed too slowly to give an economic return. In the new process, the residue is treated with hydrochloric acid to form soluble nickel chloride, and the nickel recovered by an electrolytic process. It is suggested that this new plant has been erected at the town of Pisteritz near Bitterfeld in Central Germany.

MANUFACTURE OF IRON POWDER.

Raw Material.

The raw material used for the manufacture of Iron Powder was steel scrap and spent iron pyrites from the Sulphuric Acid Plant I.G. This material is melted in a 4-ton Rotary furnace of a modified Brackelsburg type, patented by a Dr. Thomas, which is capable of both rotation and end-tilting. This furnace is heated by improved water gas (C.V. 2000 Cals./m^3) to the operating temperature and then maintained in operation by pulverised coal. When the charge is completely liquid, the furnace is tilted and the metal run into a shallow bed of sand to form layers of metal from 1" to 2" thickness.

This metal is described as Eisenstein and contains about 92 per cent Fe, 8 per cent S.

The capacity of the furnace was stated to be 15 to 18 tons per twenty-four hours.

An alternative raw material was Eisenschwamm, reduced iron, which was imported from Sweden in the form of circular discs about 8" diameter and 2" thick. This was kept as a standby for occasions when their own Eisenstein was not available.

Manufacturing Process.

1. The Eisenstein was broken into pieces easily handled and then crushed in a breaker to nut size, maximum about 10 m.m.

2. The broken metal is charged into reaction cylinders practically duplicates of the Nickel chambers and subjected to the action of CO gas at a temperature of 200° to 220°C under a pressure of 70 to 200 atmos. for four or five days. As in the case of Nickel manufacture, the chambers are operated in groups of four and each group has a capacity of about 18 tons of metal. In four to five days approximately 70 per cent of the Fe in the Eisenstein is converted into Iron Carbonyl, - $Fe(CO)_5$ - and the residue is remelted in the rotary furnace with more scrap steel. The gas issuing from the reaction chambers is cooled to condense the Iron Carbonyl and the liquid Carbonyl is trapped and

collected in the same manner as the Nickel Carbonyl.

3. The liquid Iron Carbonyl is transferred to the decomposition house. The liquid Carbonyl is distributed from a central holder into vapourising cylinders where it is heated by steam coils and the vapour passes into the decomposition cylinders. These cylinders are approximately 3 m. high by 1 m. diameter and are operated at a temperature of 240°C under normal pressure. The heating of the cylinders is by means of Kraft Gas, which is producer gas made from brown coal and has a C.V. of 1600 to 1800 Cals./m^3.

The $Fe(CO)_5$ - B.Pt. 103°C - decomposes, precipitating the iron as powder and the liberated CO is recirculated. The Iron Powder so formed contains carbon varying from 0.6 to 1.2 per cent as impurity resulting from the decomposition of the CO gas by the finely divided iron. To minimise this decomposition of the CO gas, a small quantity of dry NH_3 gas is introduced into the decomposition cylinders and the deposition of carbon limited to the above stated amounts.

The capacity was stated to be 360 to 400 Kgs. per day per unit, - with eight units in operation, 2.8 tons per day or roughly 20 tons per week.

The decomposition cylinders are cone-shaped at the lower end and the Iron Powder collects in a cylindrical chamber fitted with sealing valves above and below, and is then collected in dryms by way of a collapsible rubber sleeve.

4. The crude Iron Powder is then heated in Glühofens in an atmosphere of hydrogen at a temperature of 300 to 500°C and preferably with a little water vapour present to remove any CO gas or oxide of iron still remaining in the powder and also to reduce the carbon content. The gas content of the Powder Iron after this treatment was stated to be not more than 0.1 per cent of hydrogen and nitrogen. These ovens are horizontal steel chambers three or four m. long by 1.5 m. wide, and about 250 m.m. deep, and are heated by electric resistance coils.

5. The Iron Powder is then crushed in a Ball Mill. screened and packed for shipment.

The main uses of the product are for electromagnetic purposes, the chief consumers being Telefunken, Siemens, and the Osram Co., and for accumulator plates.

One of the main applications is for the manufacture of the well-known Puppin Cores and for this purpose the Iron Powder is pressed into the shape required.

Eisenrot.

A part of the production of Iron Carbonyl is used for the production of iron oxide of high purity for the manufacture of rubber, and as a pigment. The Eisenrot is made by burning the Iron Carbonyl with an excess of air whereby the Fe is oxidised to Fe_2O_3 and the product is deposited in an extremely fine state of division.

Further Treatment of Metal Powders.

The metal powders made by the Carbonyl process are in an extremely fine state of division, the particles having a diameter of 2 to 10 . The particles of iron are, in general, roughly spherical, but those of nickel are generally acicular. The size of the particle is governed by the conditions in the decomposition cylinder. The high operating temperature accelerates decomposition of the Carbonyl and tends towards a lighter powder, - on the other hand, a greater height of the decomposition cylinder, giving a longer distance of travel for the particle in falling to the bottom tends to produce larger particles by decomposition of more metal during the course of travel.

The packing density of Nickel Powder was stated to be 0.8 to 3.5 Kgs. per litre with a normal 2 to 3 Kgs. per litre. The packing density of Iron Powder was given as 3 to 4 Kgs. per litre.

Both Iron and Nickel Powder have been used for the manufacture of sintered articles and also for the manufacture of rolled strip, wire or hammered shapes. Both Iron and Nickel Powders are sintered by heating to a temperature of 900 to 1100°C in an atmosphere of hydrogen. In this sintering operation there is a considerable shrinkage due to the elimination of the inter-particle space, and with prolonged heating at high temperatures, the density can be made to approach that of the metal made by melting processes. The sintered material can be rolled or hammered or drawn in the normal manner, the sintered block being coated with a magnesia wash to prevent oxidation during heating and to prevent sticking.

Samples of Iron and Nickel Powder were obtained and will be reported on when the samples are received.

Nickel-Iron Alloys.

During the war there has been considerable development in the manufacture of magnetic alloys made by the sintering of nickel and iron powders. This work has been carried out by the Hereaus Co. and by the Deutsche Edelstahlwerke, the latter company being the main consumer of the electrolytic nickel made by I.G. Farben.

Other Metal Powders.

Dr. Schlechit referred to the manufacture of other metals and the research work which he had carried out on this subject. He stated that throughout the war and for some two or three years previous to the war, his sole work had been in the supervision of the Nickel and Iron Powder production at Oppau and that he had practically nothing of value to report which was not disclosed in the patents taken out by his Company before the war.

He outlined the difficulties in the formation of the Carbonyls of cobalt and molybdenum, and stated that very little of these materials had been manufactured.

He also commented on the production of powdered chromium of high purity. This metal is prepared by the reduction of Cr_2O_3 which is precipitated from a solution of chromium chloride or chromium sulphate. The reduction is carried out at a temperature of 800 to 900°C, with a pressure of 100 m.m. by dry hydrogen over a period of 3 to 4 days. The hydrogen was received from the synthetic ammonia plant and was previously compressed to 200 atmos. and dried by phosphorus pentoxide. The powdered chromium contains 99.7 per cent Cr. An important factor in the preparation of this metal for sintering purposes is the presence of oxygen which must be below 0.1 per cent to ensure good sintering properties.

APPENDIX "A".

Correlation of Buildings Plans of Ludwigshafen.

Oppau Works.

Oppau Works Plan Nos.	Nos. on I.S.T.D./C.401 Plan 3A.	Description.
46, 53	122, 123	Compressor House and Nickel Carbonyl House. CO gasholder.
217, 286 and 287	126, 127 and 128	Nickel Powder plant.
309, 310 and 311	193, 194 and 195	Iron Powder plant.
706	166	Store and Crusher for Nickel Matte or Eisenschwamm.

APPENDIX "B".

C.I.O.S. Black List Target 21/22(a).

Personnel for Further Interrogation.

Dr. H. S. Grimm	-	Director in Research.
Dr. Leo Schlecht	-)	Research in Powder
F. Duft-Schmidt	-)	Metallurgy.
Dr. Trageser	-	In charge of Eisenpulver.
Dr. W. Schubardt	-	
H. W. Gloth	-)	In charge of Gas Manufacture
	)	and synthetic NH_3, fertilisers,
Dr. Karl Goeggel	-)	etc. Oppau.
Dr. Klippel	-	Nickel Pulver.
Dr. Pfleiderer	-	Research.
Dir. Brendel	-	Ludwigshafen Headquarters.
Dr. Fahrenhorst	-	Works Manager.
Dr. Wildhagen	-	Chief Engineer.
Dr. Haptrecht	-	Assistant "
Dr. Naumann	-	Engineer.
Dr. Fraozer	-	"
Dr. Braun	-	"
Dr. Zimmermann	-	"

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