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HIGH TEMPERATURE REFRACTORIES AND CERAMICS

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

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HIGH TEMPERATURE REFRACTORIES

AND CERAMICS.

Report by:

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BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE.

HIGH TEMPERATURE REFRACTORIES AND SPECIAL CERAMIC MATERIALS.

Reported by: F/L. W. Watt, R.A.F. (M.A.P)
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OBJECT.

The object of B.I.O.S. trip number 1252 was to study the manufacture of high temperature refractories and ceramic materials and obtain information on the fabrication of ceramic turbine blades.

1. HERMANN GOERING LUFTFAHRTFORSCHUNGSANSTALT VOLKENRODE, NEAR BRUNSWICK.

(a) DATE OF VISIT.

The works was visited on the 2nd and 3rd October, 1945. The personnel interviewed were: Professor Dirksen, Herr Trapp and Herr Bamment.

(b) INTERVIEW WITH PROFESSOR DIRKSEN AND HERR TRAPP.

Dr. Dirksen had been in charge of experiments on the mechanical properties and thermal shock resistance of various refractory ceramic products. He briefly outlined the substance of five reports which appear as Appendix I.

The most important discovery was that although the compressive strength of the ceramic materials tested was considerably greater than the tensile strength at room temperature, the two became approximately the same at about 700° to 800°C.

The apparatus used for this experimental work was exhibited by Herr Trapp. Tensile tests had been largely abandoned due to the difficulty of obtaining true axial loading of the specimens. Instead measurements of transverse strength were carried out in a horizontal electric furnace heated by sillit rods. Provision was made for measuring the extension of the portion of the test rod in tension and the contraction of that part in compression by means of two mirror systems and thus testing the similarity of the stress-strain curves for tension and compression. Creep tests were carried out in this apparatus in addition to measurements of strength. In vertical compression tests the deformation on opposite sides of the specimen was measured in order to ensure correct alignment of stress. The apparatus was orthodox in other respects.

Tests had also been done on a comparative thermal shock characteristics of ceramic venturis, presumably to ascertain their suitability for combustion nozzles in gas turbines and for rocket propelled projectiles. Combustion Gases at an estimated temperature of $1700^{\circ}C$ and approx. 30 atms pressure issued through the venturi for 5 mins. The cycle was repeated until fracture. The results were poor, most of the ceramic venturis failing by cracking after one or a few cycles. Cutting the venturi into three equal segments and mounting them in a metal case, with or without the backing of another ceramic venturi was found to prolong the life.

(c) <u>INTERVIEW WITH HERR BAMMERT</u>.

Herr Bammert of the engine division was interviewed on the application of ceramics to gas turbines. Blades in various ceramic materials such as sillimanite, alumina, porcelains, etc. which had been made by the following firms were inspected.

Stemag	-	Berlin.
Hescho	-	Hermsdorf.
Degussa	-	Frankfurt.
Koppers	-	Dusseldorf.

State Porcelain Factory - Berlin.
Siemens Halske - Berlin.

No actual turbine had been built with ceramic blades; the work had been only on methods of mounting and on heat resisting properties. A turbine with metal rotor blades and ceramic stator blades had been designed. The mounting of the blades had presented considerable difficulty. The most suitable method was found to be sintering the blade in a slot in a heat resisting steel base with powdered iron. The sintering temperature was 1300°C., the various operations being normal powder metallurgy technique.

The heat resisting properties of the blades were tested in a combustion rig in which they were subjected to a stream of hot combustion gases, the mass flow being of the same order as in a gas turbine, and to various degrees of temperature change, until the blade failed by cracking. The outstanding blade in this test was one consisting of a mixture of silicon carbide and clay with a glazed surface.

One blade, made of sintered alumina, was hollow to permit of hot air being passed through it to mitigate the thermal shock of combustion gases impinging on the exterior. This was considered a promising development.

It was stated that the intention was, had the war proceeded, and conditions allowed, to build a turbine with a water cooled metal rotor and a glazed silicon carbide stator and to arrange for an unequal reaction between stator and rotor so that the metal was more stressed than the ceramic material.

(d) CONCLUSIONS.

It is difficult to assess the value of the work done at Volkenrode. The different divisions of the experimental work seemed to have proceeded independently. For example, Professor Dirksen was not informed of the composition of the bodies he tested, and the engine section proceeded on the assumption that ceramic materials are weaker in tension than compression when hot, despite Professor Dirksen's experiments. The German workers conveyed the impression that they considered that there was a fair prospect of ultimately providing a method of using ceramic materials in gas turbines.

2. SCHEIDHAUR UND GIESSING, DUISBERG (DIDIER-WERKE, A.G.)

(a) DATE OF VISIT.

A visit was paid to this factory on the 8th October, and Herr Katheriner, the works manager, interviewed.

(b) CONDITION OF TARGET.

The works were badly damaged in parts, other parts were intact. Some of the kilns, pressing shops, and carbon brick kiln were badly damaged. All the grinding and mixing plant was intact. Roughly 25 per cent of the plant was damaged but a considerable output was thought to be possible with the factory as it stood.

(c) PRODUCTS.

The works used to make firebricks (chamotte bricks), silicon carbide products, glass tank refractories, sillimanite products, corundum refractories, graphite stoppers and nozzles, carbon blocks and acid resisting bricks. Total month output 4,000 tons. Sillimanite bricks had not been made during the war owing to the cessation of imports of kyanite. Highly grogged fireclay bricks were made as substitute. The methods of manufacture for most of the products were very similar. We directed our attention chiefly to the sillimanite, silicon carbide, corundum and carbon products.

(d) MANUFACTURING PROCESSES.

The works is equipped with a number of overhead conveyors to deliver raw materials from each main dump to the jaw crushers, from which it passes to the crushing rolls; it is then magnetted, elevated, screened in rotating octagonal sieves or double banks of vibrating sieves operated mechanically. Dust collection by the Beth system (Lubeck) is provided. The fine middle and coarse fractions are conveyed on the same vibrating conveyor belt side by side and can be diverted as required to bunkers collecting the required grade of material. Several openings to different bunkers are provided. Each bunker is equipped with a weighing machine so that each item of the dry batch can be discharged on to a moving belt which in turn discharges it into a receiver on an overhead rail. Two types of mixer were seen, the Werner and Pfleiderer and the Eirich, the latter gradually displacing the former in this works. After 15 minutes dry mixing the slip required to effect the bonding is introduced. This consists of Witterschlik clay, dried and ground in a ball mill with iron or flint pebbles (according to type of mix) and mixed with water and 0.5 per cent Kasseller Braun, 1.5 per cent dextrin, 1.2 per cent soda ash in a blunger. After 15 minutes vigorous mixing the slip is stored in an arc for 2 or 3 days with slow stirring. After further mixing of slip with this dry batch in the Baker Perkins or Eirich mixer, it is delivered into a waggon on the floor below to be taken to the dry pressing or tamping appliances.

The composition of typical mixes was given as:-
Sillimanite Mix. 2½ - 6 mm. calcined kyanite 33 per cent; 1 - 2½ mm. calcined kyanite, 17 per cent; 0 - 1 mm. calcined kyanite, 50 per cent; fine clay added 8 per cent. water content, 5 - 6 per cent.
Corundum Mix. Similar to the sillimanite, replacing the calcined kyanite by fused alumina similarly graded, obtained from Feldmühle.

Silicon Carbide Mix. 1 - 2 mm., 50 per cent; 0 - 0.4 mm., 10 per cent; 0 - 0.1 mm., 40 per cent; 8 per cent of added clay.

For moulding stock silica brick sizes a rotating table type press in which the damp mix is swept in turn into each mould and pressed from below by eccentric pressure action is used. The output was given as 6,000 bricks in 8 hours. A hydraulic press employing 400 atmospheres on the ram and 277 kilo per sq. cm. on the top and bottom of normal sized bricks was seen. The ram presses 3 normal sized bricks per stroke, 1,000 bricks per stroke per hour can be shaped. To obtain the pressure water is pumped to an accumulator at 50 atmospheres pressure; this can be intensified to 400 on the ram of the machine.

The tamping apparatus and method for which this firm is noted was explained by reference to both small and large shapes. The mixture is tamped into the mould using a pneumatic ram with a curved end measuring about 3 in. x 1½ in. having several teeth. For the larger pieces one man continuously feeds the mixture and another continuously tamps. Pieces measuring about 4 ft. x 6 ft. x 6 in. take about 1 hour to make using two men. The moulds are carefully designed of metal so as to be readily disconnected. The process is illustrated in Trans. Ceram. Soc., Wedgwood Bicentenary Volume, 280, 1930 and in "Refractories" by F.H. Norton, McGraw Hill, 2nd Edition, 1942, pp.245 T 246. Drying of the shapes takes place in a building of several storeys, hot air being passed up through the whole building from a heater at the base.

The firing of the products (other than carbon or graphite) takes place in a small Hoffmann type of continuous kiln with permanent division walls. The dimensions of a chamber are about 8 ft. x 8 ft. x 7 ft. (to the crown). Three chambers out of the 16 are under fire at one time. Firing is to Seger Cone 13, 14, or

15 according to the ware being fired. The crown above the firing zone is of silica bricks.

For making carbon blocks (for blast-furnace hearths) foundry coke of low ash content is used. It is crushed in an edge runner, screened, and mixed in a very strong Baker and Perkins machine at 60° - 70°C. with tar. The mix consists of 84 per cent coke 0 - 4 mm. in size, and 16 per cent tar containing 75 per cent anthracene oil and 25 per cent pitch. After mixing it is discharged on to a warm floor and allowed to cool slightly and tamped or pressed while still warm. The press is a large hydraulic one, the dies are not heated. The operation is similar to the one already described. The blocks are fired, buried in coke dust in four parallel trenches about 4 ft. deep and 4 ft. wide and 150 ft. in length which are heated from the sides by 6 rows of step grate combustion checker work flues, which form the walls of the trenches. Fine coal is fed on to the grates and burnt by hot air drawn along the flues. The coal is thus burnt much in the same manner as a Hoffmann kiln. The fire is made to progress round the circuit, the flue gases eventually passing to a main central flue and to the stack. The temperature attained is a red heat. Three weeks are required to reach top temperature and 3 weeks are allowed for cooling. The coke in the trenches is not covered. Heavy blocks are removed by means of blocks and tackle. Smaller sizes are removed by hand. The blocks are then ground to size, assembled, made to fit, and numbered for reassembly on the site.

The mix for graphite stoppers and nozzles consists of 50 per cent chamotte 0 - 4 mm.; 25 per cent clay 0 - 0.5 mm.; and 25 per cent of Bavarian graphite 0 - 4 mm. This is made into a plastic mass and shaped in hand moulds.

(e) CONCLUSIONS.

The works produced some very attractive examples of tank blocks of sillimanite and chamotte and of silicon carbide for zinc retorts. The accuracy of shape obtained by the S. u B. process originated by this firm was well-known before the war. The works was not particularly well laid out but good use had been made of conveyor systems. Owing to the obvious heat losses the firing process cannot be regarded as efficient.

3. HEINRICH KOPPERS, DUSSELDORF. HEERDT.

(a) DATE OF VISIT.

This works was visited on the 9th, 10th and 11th October, 1945. The personnel interviewed were:-

Herr Schuffler	(Technical Director).
Dr. Frank	(Works Manager).
Herr Kuhn	(Chemist).

(b) CONDITION OF WORKS.

The works was largely undamaged. There was only sporadic damage by artillery and some internal damage and looting by displaced persons.

(c) PRODUCTS.

These may be classified as follows:-

(1) Firebricks.
(2) Silica Bricks.
(3) Magnesite and chrome-magnesite products
(4) Special refractories: sillimanite, silicon carbide, and corundum products.
(5) Sillimanite and sintered alumina laboratory ware.

Groups (1), (2) and (3) had been investigated by a previous team. Attention was paid to group (4) and particularly group (5).

(d) INSPECTION OF PLANT.

Koppers specialize in high quality sillimanite products. All the kyanite is prefired in a large tunnel kiln of cross section approximately 6 ft x 6 ft. This kiln is fired with producer gas made on the site and has a regenerative system for preheating the air. Silica bricks are used for the lining and the temperature of firing is 1500°C. During the war chamotte or chamotte fortified with alumina had to be used.

The manufacturing process consists in crushing the calcined sillimanite, magnetting, elevating to a 3 tier shaker sieve and collecting the required grain size fractions. A tube mill is provided for fine grinding. Mixing is carried out in an Eirich mixer following practice similar to the S. u G. method. Various proportions of clay may be added. For best quality ware selected sillimanite and only 5 per cent of clay dispersed with Kasseler Braun is used. Wooden moulds, steel-faced, are used for the tamping process. A pneumatic hammer is used for tamping one man working alone for small pieces, roughening the surface before making each fresh addition. Burner blocks for Wistra burners are made by this method using a mould which separates at the junction of the cones. For high temperature furnaces these blocks are made with a mixture of sillimanite and corundum to give an alumina content of 85 per cent. The water content of the tamping masses is 3 - 4 per cent. A vibrating mould hydraulic press is also available based on a design used for making moulds for foundry work. The mould is vibrated while a pressure of 70 atmospheres is applied.

Ware is dried on steel pallets in a Proctor dryer.

The kiln usually employed for firing the sillimanite and special goods was about 230 ft. long. The kiln was about 4 ft. wide and stacked 4 ft. high, fired by Wistra (Wirbelstrahl brenner) burners using coke oven gas and air preheated by recuperation in heat resisting steel and silicon carbide pipes. The temperature of firing is up to 1600°C. Silica bricks are used to line the high temperature zone.

There are several intermittent gas fired kilns for firing sillimanite ware to 1600°C. The goods are introduced into the kiln on a truck measuring about 8 ft. x 12 ft. and stacked about 8 ft. high. Heating is from either side using Wistra burners. Hot air from a cooling kiln adjacent had sometimes been employed for combustion but preheating within the furnace had been discontinued during the war owing to difficulties in obtaining heat resistant steel. The furnaces are lined with 60 per cent porosity sillimanite bricks of their own manufacture. The condition of these bricks was good despite the temperature at which they had been used. The bricks are made of hand-picked calcined kyanite, the grading being 70 per cent 0 - 0.5 mm. and 30 per cent 0 - 0.1 mm. Nine parts of the graded kyanite and one part of Pfelzer clay are mixed. To promote porosity a semi-bituminous coal of only 4 - 6 per cent ash and low swelling properties is added being graded so as to correspond to the grain size of the kyanite and added in an amount which may reach up to 14 per cent of the total weight of the mix. The mass is hand moulded or pressed in a hydraulic press with a limited travel. Firing is normally to 1450°C., but the super quality bricks to withstand 1600°C. on the hot face are fired to 1600°C.

A number of gas fired furnaces for firing alumina and sillimanite to 1800°C. were inspected. They are of circular cross section, diameter 2 ft., internal height 10 ft. Heating is by two diametrically opposed vertical rows of Wistra burners. Alumina and sillimanite tubing up to 2 metres long is fired in these furnaces suspended from a "pipe-rack" slot arrangement in the roof.

This furnace is lined with special quality sillimanite bricks made from hand-picked kyanite bonded with 5 per cent clay. The maximum grain size is 5 mm. and the grading is selected to give close packing. Between 20 and 25 per cent of sillimanite ground to less than 6 μ is included in the mix. Shaping is by pneumatic hammer and the firing is to 1600°C. in a 72 hour cycle. A detailed drawing of the furnace is available on application to the authors.

(e) PROCESSES FOR SILLIMANITE AND ALUMINA WARE.

Sillimanite Ware.

The raw kyanite is fired to 1500°C. for 24 hours then ground by normal methods to a maximum grain size of 3 mm. Further grinding is done in a ball mill with flint pebbles, a typical charge being 1,500 kgm. fired kyanite to 900 litres of water with the mill half filled with flints. The mill is run at cascade speed for 2 days giving a total of 15,000 revolutions. This reduces all the particles to less than 6 μ. The ground slip is then strained through a 10,000 mesh/cm^2 sieve into a mixer in which 1,000 kgm. Pfelzer clay and 500 kgm. kaolin are added. After thorough mixing, passing over a wet magnet and straining through a 2,500 mesh/cm^2. vibrating sieve the slip is filter pressed and the mass aged for 6 months in a cellar. Before moulding the mass is kneaded by machine for three hours to remove air and then is ready for extrusion. Extrusion is vertically downwards, the tubes being caught on a V-shaped board held at an angle, and pinched off when the desired length has been attained and the extruded tube rolled out on to a flat board. For sizes 1 - 7 mm. in diameter a hand operated press with a simple die plate affixed to an untapered cylinder is used. For larger diameters a motor driven press of similar design with a variable gear coupling is used. The tubes are dried in air for 1 - 3 weeks according to size. If closed ends are required they are affixed while the tubes are in the leather hard state. The rounded end is either slip cast or jolleyed to shape if over 60 mm. diam. The flange for supporting the tubes for firing in the kiln was also affixed at this stage. This was done by cutting off a length from a tube of slightly larger size and making a union with slip. The small sizes were merely deformed at the top and a ring of the body bent round so that the piece could be slung in the "pipe-rack" suspension brick in the roof of the kiln.

In addition to shaping by extrusion, suitably shaped articles are pressed, slip cast or jolleyed. For slip casting 0.5 per cent of water glass ($1Na_2O:1SiO_2$ 34° Baume) is added to the slip. As the required water content is 23.0 per cent (on the dry weight) it is necessary to add dry material to the usual slip from the blunger. Small crucibles below 60 mm. in diameter are slip cast and larger sizes jolleyed in plaster moulds.

Ordinary sillimanite ware, called 101 is fired up to 1600°C then held at 1600°C. The firm consider this grade equal to pythagoras porcelain. Special quality sillimanite ware, 91, made from hand picked kyanite but other-wise processed as above is fired for 2 hours at 1780°C. This ware is gas-tight up to 1500°C. as compared with 1400°C. for 101. The shrinkage of the 91 sillimanite on firing is 15 per cent.

Alumina Ware.

The raw material is a hydrated alumina of approximate composition $Al_2O_3.3H_2O$. This is wet ground for 3 - 4 hours, filter pressed, briquetted and calcined at 1500°C. for 12 hours. The Na_2O content after calcining is about 1 per cent. After calcining the material is crushed and ground in a rubber lined ball mill. Mill charge is 300 kgm. alumina and 150 litres water.

The grinding is done by hard-fired sintered alumina plates roughly 10 cm. x 10 cm. x 2 cm. with which the mill is half filled. The grinding period is 2 days at 40 revolutions per minute after which all the particles are less than 5 . The slip is then filter pressed and the material air dried for 8 days.

For extrusion a bond made as follows is used:- 600 gm. of a hydrated alumina, approximately $Al_2O_3\ 1.5H_2O$ obtained from Lautawerke Saxony is treated with 800 cc. of hydrochloric acid (4 parts of concentrated acid diluted with 1 part of water). Then cold water is slowly run in with continuous stirring until the total volume is 3 litres. Heat is developed and on cooling the mass sets to a stiff gel. 25kgm. of this gel is mixed with 100 kgm. of dry ground alumina in a rubber lined Werner and Pfleiderer mixer and the mass adjusted to the desired extrusion consistency by the addition of water.

A suitable amount is then cut off and placed in a simple evacuation chamber which it fits fairly closely. It is compressed while under vacuum and then transferred to the extrusion cylinder. Handling methods follow those for sillimanite ware. Ordinary hardened steel dies are used. Except for the smaller sizes the ends of closed tubes are slip-cast and jointed to the open tubes by slip when both are quite dry. Jolleying methods are used for suitable shapes.

For slip-cast alumina ware a slip is made from 100 kgm. ground calcined alumina, 45 litres of water and 5 litres of hydrochloric acid (4 parts of concentrated acid to 1 part of water). Normal slip-casting procedure is followed. 800 mm. is the greatest depth used in casting. Tubes above 48 mm. diam. are slip cast.

The alumina ware is fired for 2 hours at 1820°C., the temperature being reached in 6 hours. The furnace is allowed to cool overnight. A technique for suspending the alumina tubes similar to that used for sillimanite is employed. Small ware is fired in saggars made of alumina. The shrinkage is 23 per cent from the mould size. No additions are made to restrain the grain-growth during recrystallization. The largest grain size in their ware is stated to be about 0.1 mm.

(f) CERAMIC TURBINE BLADES.

Koppers made a few ceramic blades of sillimanite for the Hermann Goering Luftfahrtforschungsanstalt. The blades were made by extrusion through a die having an obstruction plate on the inside which could be adjusted by trial and error to give equal rates of flow over the different blade thicknesses.

They had no knowledge of the suitability of sillimanite as a material for gas turbine blades.

(g) CONCLUSIONS.

Koppers enjoy a high reputation for their special refractories. The good performance of their special quality sillimanite bricks may be attributed to the careful selection of the sillimanite and its grading and the use of minimum amounts of clay. The kiln for calcination of raw kyanite is inferior in design to one used for this purpose in this country.

In the manufacture of their laboratory alumina ware the method of obtaining a plastic gel appears to be a special development of this firm. Degussa (Frankfurt) for example, introduce organic combustible material such as gum tragacanth or rubber. The care taken to avoid contamination of the alumina with iron during preparation of the mixes is noteworthy as it avoids the need for subsequent acid washing treatment. A high standard of craftsmanship is attained.

4. FELDMÜHLE, LÜLSDORF/RHEIN - MANUFACTURERS OF FUSED ABRASIVES AND REFRACTORY MATERIALS.

(a) DATE OF VISIT AND PERSONNEL INTERVIEWED.

The works was visited on the 12th October, 1945, and the following members of the staff were interviewed:-

Dr. Holsch, General Manager,
Herr Hack, Chemist,
Herr Schmidt, Engineer.

(b) CONDITION OF TARGET.

The works was very little damaged; a small amount of structural damage due to shelling had been suffered. The management were waiting for material to repair buildings and for permission to restart manufacture.

(c) PRODUCTS.

The firm manufactures a considerable range of chemicals, including the following fused refractory materials in which we were interested.

(1) White fused alumina (Trade name BIKORIT).
(2) Pink fused alumina (Trade name DIAUBIN).
(3) Brown fused alumina (Trade name REDURIT).
(4) Black fused alumina (Trade name [illegible]).
(5) Fused magnesia.
(6) Fused spinel.
(7) Fused ferro-silicon (by-product).

The above products are fused on a large scale and afterwards crushed and graded for sale to the abrasive, and refractory industries.

(d) MANUFACTURING PROCESSES.

A special feature of the plant is the degree of mechanisation and the continuity of operations.

(1) White fused Alumina (BIKORIT).

Handling and Furnace Feeding Arrangements.

The raw material used is Baeyer processed calcined alumina from Martinswerke, Birkheim. A very complete assembly of cyclones and backing pump of the Nash type is used to transfer the raw materials to the various hoppers. The material is sucked from the trucks into the storage hoppers and as required material therefrom is hand-fed to a suction point or sump at ground level, and elevated to the main hopper above the furnace. The return air flow passes through two cyclones and a water filter before returning to the pump, and escape material from the first cyclone returning to the furnace, this amounting to about 0.3 per cent of the charge weight. The material from the main hopper passes from a worm conveyor to a tipping scale pan, delivering batches of about 40 lb. at a time, direct to the furnace through twin delivery shutes. (See diagrammatic sketch No.1.)

Furnace.

The furnace consists of a slightly conical steel cylinder 180 cm. (6 ft.) in diameter by 180 cm. (6 ft.) in depth cooled with a water spray mounted on a base with rail wheels fitted. During fusion the assembly is rotated on a rail turntable, actuated by a reduction gear and pawl rod which engages a rack on the periphery of the base plate. The furnace rotates once every 4 hours thereby producing a symmetrical fusion (see sketch No.2).

The arc rods are vertical and held in water cooled plate electrode holders, suspended from steel beams by a pulley system from an overhead crane. The running conditions are as follows:-

Power .	Single phase supply from a 550 K.V.A. Transformer.
Carbon Rods.	Rectangular 40 xm. x 20 cm. at 90 cm. centres.
Average current.	About 2,000 amps.
Voltage between electrodes.	Approx. 170 - 220.
Arc Starting.	Coke bed, about 60 lb. in weight arranged between electrodes.
Total running time.	50 hours.
Total charge weight.	About 15 to 16 tons.
Fusion weight.	About 14 tons.

Processing.

After cooling by water spray, the conical furnace body is withdrawn and the fusions lifted by crane and dropped onto a steel breaking spike and thence to two stages of jaw crushing, (the jaws are of 12 per cent manganese steel). The product is then passed through manganese steel rollers over a drum - type magnetic separator, and thence to a multi cascade horizontal screen bank of the oscillating type, giving grades from ¼ in. mesh downwards. Finer grades for polishing powders are dealt with in a separate 10 stage vertical sieving bank of the oscillating type and final products fall to bagging hoppers. The grain sizes are those normally required by the abrasive industry.

Fine milling of the fused alumina is carried out in three cylindrical ball mills about 2 ft. 6 in. in diameter and 6 ft. in length with liners and balls of 2 per cent chrome steel. They are connected to a cyclone air separator of the Hardinge type. The direct product from the mill is approximately 30 microns and finer, with a small percentage up to 50 micron size. The air separated material is predominantly up to 40 microns. The iron content is 3 to 4 per cent and is not normally removed. Acid treatment is carried out on a small scale if specially required.

(2) Pink Fused Alumina (DIRUBIN).

The material is produced in a similar manner to the white fused alumina. An addition of 0.5 per cent of chromic oxide is made to the melt.

(3) Brown and Black Fused Alumina (REDURIT and NORMALIT).

Raw Materials.

Bauxite is obtained from Marseilles, France and Hungary. Anthracite is mixed with the charge to reduce the iron oxide. The ferro-silicon formed as a by-product contains 16-18 per cent silicon.

Furnace.

The furnace body is of similar design to the furnace for making white alumina but is larger, being about 240 cm. (8 ft.) in diameter by 190 cm. (6 ft. 3 in.) in depth. It is rotated at a speed of about 1 revolution per 6 hours. The running conditions are as follows:-

Power: Three phase supply from a 2,000 KVA transformer.

Carbon Rods: Three rods 55 cm. (22 in.) in diameter are set vertically at 120 cm. (4 ft.) centres; sections are joined together by means of a heavy male and female cone thread. The total length of the rods is about 8 ft.

Electrode Holders: The electrodes are held by 6 sectional water-cooled clamps secured by bolts passing through an external peripheral ring which is attached to the lower end of the main metal sleeve supporting each of the suspended electrodes. The ring is also water cooled by by-pass water. These heavy rods are supported by a wire rope pulley system passing to heavy counterbalance weights, and elevation is carried out by a crane above.

Voltage at Electrodes: About 150 volts between phases.

Arc Starting: Coke bed used to bridge the 3 rods.

Total Charge: Bauxite, About 30 tons.
Anthracite Coal. " 2½ tons.

The charge is mixed and fed in by hand from a large steel platform arranged at the level of the furnace top. An operater breaks down the shell formed progressively during the fusing, by means of a steel bar. A head of about 3 in. of material is maintained above arc level, and periodic charging occurs every 15 - 20 minutes.

Total Running Time : 60 hours.

Total Weight of Fusion: About 22 tons.

Useful Weight of Black Fused Alumina: About 13 tons.

The balance consists of outer coating eventually refused, and the by-product ferro-silicon.

Processing.

The fusions are impact broken and jaw crushed, passed through rollers and over drum magnetic separator and thence to multi cascade horizontal bank of screens of the oscillating type, the resulting products passing to bagging hoppers.

In the case of the brown bauxite (REDURIT) the sequence follows direct to a rotary calcining oven, coal fired to 1200°C. The feed is continuous, and the final stage of the series of operation consists of screening of finer meshes on a special type of sieve designed by the firm, the material being lifted by elevator to the upper hoppers. The special feature about this screening bank is that it introduces a "side" oscillating motion to the screens which are angled in the normal way to the horizontal, resulting in a zig-zag path of movement of the particles giving a high efficiency factory

Each of the Four cascade banks used, consists of two vertical tiers of three sieves linked together, and each bank is suspended by pine leaves to allow transverse motion. This motion is imparted by a floating rotary steel shaft having eccentric sheaves and horizontal rods linking the sieve frame at each end. The shaft is suspended from above at the two bearings by pine leaves, and a motor drive above transmits motion by belt to a pulley in the centre of the shaft, the whole assembly producing a highly efficient oscillating motion. This is a definite improvement on the normal and oscillating type, and will be of interest to screening plant manufacturers.

The products pass to 24 bagging points, giving grain sizes of the normal standard grades required by the abrasives industry.

(4) Fused magnesia.

Calcined magnesite obtained from Jugo-Slavia or Greece is fused in a similar vertical conical furnace, the size of the body being similar to the furnace for fusing the white alumina (6 ft. diameter x 6 ft. deep) but fed from the platform in a similar manner to the black bauxite furnace.

The supply is single phase taken from a 1,500 KVA transformer, and very large carbon electrodes are used, namely 60 cm. (24 in.) x 35 cm. (14 in.) rectangular section, set to hang at about 25 cm.

(10 in.) apart, and suspended by steel plate electrode holders, (water-cooled), which in turn are suspended from horizontal steel beams which also support the heavy leads. These beams are supported at two points by a rope and pulley system actuated by a crane, so that a horizontal pull on one rope elevates the beam as required.

Very little fused magnesia has been made, and this furnace had been used for making smaller fusions of bauxite. No further running data were available, but it was estimated that fusions of about 12 tons were produced in 45 hours total running time. These large fusions of magnesia cool slowly, and thereby produce large crystals of periclase in the centre; specimens which were seen measured 1½ in. to 2 in. by ¾ in.

(5) Fused Spinel.

The materials used for the manufacture of fused spinel ($MgO.Al_2O_3$) are the white calcined alumina and the calcined Jugo-Slavian magnesite. The magnesia furnace is used. Relatively small amounts of fused spinel had been produced.

(6) Final Grading of Finished Products.

As a safeguard against any faulty screening in the normal processing, three small plants, each consisting of two bank oscillating sieves combined with drum or roller type magnetic separators are provided. Each bag of material is elevated on a bag hoist to one of these units and any oversize or magnetic ingredients removed. The bags are then stencilled with a material and grain-size marking.

(8) CONCLUSION.

This works has been carefully laid out for the large scale production of fused alumina and magnesia. Great pains have been taken to ensure maximum efficiency in manufacture and a uniform series of products.

5. HERAEUS QUARZGLAS GESELLSCHAFT, HANAU

and QUARZGLAS FABRIK

(a) Date of Visit 17th and 18th October, 1945.

Investigator: Mr. R.B. Miller

Interviewed Dr. H. Mohn, Works Manager and Engineer

(b) General Report

The Works have been badly damaged. In the sections producing Rotosil (opaque Silica) tubes and pipes, the furnaces, of which they had two, have been badly damaged, but they are repairing parts with the intention of getting one into operation. They still have a number of steel furnace tubes ranging from about 5" to 16" diameter.

The section producing transparent quartz has suffered most, all the forming furnaces having been destroyed, including the furnaces for building up tube billets (ordinary and sub-normal qualities) and also their optical quartz furnaces. Their quartz powder preparing plant, although dislocated, could be put into commission fairly readily. They have a billet redrawing furnace, operated by one man, and are at present drawing tubing, both translucent and transparent from rather poor quality stock billets which had been normally rejected before the Works were put out of commission.

In the blowpipe section, they have about 8 men working, chiefly producing small electric heaters, for boiling tea and coffee, from translucent silica tube with internal element of nickel chrome. Other articles include transparent quartz crucibles for coking tests. They are, however, erecting suction plant (SEE SKETCH NO.1) in another room which will house about 50 blowpipe hands for the production of quartz lamps, etc. The department normally producing these lamps has been totally destroyed.

They have also in operation a small silica moulding furnace for forming opaque crucibles and basins on a limited scale.

They normally employed –	pre war	170
	during the war	250
	at present	40

Estimated damage –	furnace sections	90%	damage.
	fabrication "	75%	

The following is a list of their various products:–

TRANSPARENT FUSED SILICA

(1) Electrosil Quality

This vaporising process was not mentioned or known at the time of the visit, but is referred to in C.I.O.S. Report No. XXVIII-67 (Item No.21).

(2) "HOMOSIL" Optical Quality.

This is their best Optical Quality and is used for special blocks, lens, prisms, being relatively free from internal striations, bubbles and strains. End reflecting mirrors for range finders are made from this material.

(3) Normal Quality (SORTE I).

Chiefly tubing and apparatus made therefrom; also plates and other solid shapes for use where Optical Quality is not necessary.

(4) Sub-normal Quality (SORTE 2).

Being chiefly poorer quality rejects from normal quality manufacture, and is sold at reduced prices.

(5) Quartz Wool.

For high temperature insulation.

NON TRANSPARENT FUSED SILICA.

(1) "ROTOSIL" Tubes and Pipes, and apparatus made therefrom.

(2) Moulded Articles such as basins, crucibles, etc.

The following deals individually with the various products normally produced.

(d) TRANSPARENT FUSED SILICA.

(1) Electrosil Quality.

No data obtained.

(2) "HOMOSIL" Optical Quality.

Selected Brazilian Quartz Crystal is acid treated with cold hydrofluoric acid, and water washed with tap water and twice with distilled water, and dried. It is then passed through a rotary silica tube furnace running at about 900°C., the furnace being about 6 feet long by 5" bore, and the emerging crystal is quenched in cold distilled water to disintegrate it into readily friable pieces.

After drying, it is passed to a cone grinder consisting of a stationary quartz crystal about 12" diameter x 8" - 9" high, having a vertical hole 3" diameter, terminating in a 60° cone bored at the lower end. In this cone rotates an agate cone with provision on the driving mechanism to adjust the gap as required.

The setting preferred gives a final product 1 mm. size and below, and the powder is transferred to a simple silica pipe air separator which has the suction adjusted to remove the fines below 0.2 mm. The powder is then stored in glass bottles and is ready for fusing in the optical furnace.

Optical Furnace.

This consists of a special oxy hydrogen blowpipe with a quartz funnel attachment permitting the powder to be entrained in the flame. The powder feed is adjusted by means of the variable speed of a rotary angled silica tube above the funnel, driven by a small motor drive with variable disc traverse, and the preferred feed is about 280 gms. per hour. The burner impinges on a horizontally rotating and opposed quartz rod, this taking place inside a silica pipe economiser. The fused powder builds up within the zone of the oxy hydrogen flame, air being relatively excluded. The billet progressively formed is receded by a traverse mechanism, thereby permitting billets up to about 20" long to be made, the popular diameter being 40 to 50 mm. diameter. With 2 blowpipes side by side, a billet about 80 mm. diameter can be produced, but the quality is not so reliable.

The billet so formed can be cut direct for discs, or alternatively forged up to larger diameters in a multi blowpipe furnace, and finally moulded in graphite moulds to the required shape.

End reflector mirror plates are formed in this manner, and finally machined with carborundum cutting and grinding wheels to the desired cross section. These blocks are sent to Zeiss, who select and cut where required to produce the best quality active mirror plates, and the bases from any material considered inferior quality. After return, these plates are mounted in a special two part annealing furnace (SEE SKETCH NO. 2) having three side plungers externally spring loaded which hold the plates and base assembly in position against a jig prism mounted in the centre. The whole assembly stands on a smooth silica plate mounted on fused quartz granules in the lower half of the furnace, leaving the lug portions of the side plates exposed for fusing to the base, which is done using optical quartz rod filling and an oxy hydrogen blowpipe. Protection plates of quartz about 10 mm. thick are placed outside the active plates and on top of the jig prism to prevent flame impingement during fusing. The lower portion of the annealing furnace is kept at 860°C., and after fusing, the top portion of the annealing furnace is put on, and raised from an initial temperature of about 860°C to 1060°C and the assembly is then cooled slowly, the whole process taking about 4 hours.

(3) Normal Quality (Sorte 1)

This quality is produced in billet form either solid or hollow, in a special multi-blowpipe furnace, consisting of 4 jets of oxyhydrogen passing through the side of an opaque silica pipe horizontally disposed, which forms an economiser. This is mounted inside a water cooled steel sheet furnace housing having suction exhausting arrangements fitted at the top. (SEE SKETCH NO. 3). A special sliding platform is arranged through the side, allowing feeding on to a graphite slab, of quartz powder of from 1 to 3 mm. size, this bed being placed in front of the open end of the silica pipe. The fusion is built up initially, on a part quartz billet mounted on the end of a silica working tube, and the operation consists of progressively heating the billet and rolling the softened surface in the quartz powder. For hollow billets the bore is kept open by pushing the hot billet on to a tapered graphite pin outside the furnace.

Billets are produced up to about 24" long and 3½" external diameter, and may be further forged as required, or re-drawn down for tubing of varying sizes from 3 mm. to 30 mm. external diameter in a special furnace.

Redrawing Furnace

This furnace consists of a headstock holding and rotating a billet mounted on a ground silica tube, which is advanced progressively into the heating unit, which consists of a heat resisting steel burner giving an internal ring or slit frame, arranged for burning either oxy-hydrogen or oxy acetylene, the former being preferred. The tubing is drawn along the 50 ft. bed of the furnace on a rotating carriage mechanism, with a motor drive synchronous with the headstock drive, thereby allowing the drawn down tube to be rotated at the same speed as the billet. The speed of withdrawal of this carriage controls the size of the tubing, and this is arranged by a motor drive at the end of the bed, linked to a variable speed gear actuating a chain drive along the bed, to which the carriage is engaged when required, all controls being operated at a panel adjacent tothe burner.

(4) Sub-normal Quality (Sorte 2).

This material is chiefly supplied from the poorer specimens selected from normal quality production, and therefore follows the same technique of manufacture. Rejected crystal not normally used for either (2) or (3) is acid treated and can be processed in the normal manner thereby economising in raw materials costs.

(5) Quartz Wool.

This is produced on a special machine, (SEE SKETCH NO4) and has been supplied in large quantities for high temperature insulation mats. It consists of a carriage driven along a steel structure bed by a motor drive. On this carriage are clipped in a fairly close spaced row, 20 horizontal quartz rods or tubes, the latter preferred, about 6 to 7 mm. diameter. Each rod passes through a graphite guide and over a vertically disposed oxy hydrogen burner which reduces the fibres to about 0.2 mm. diameter. These fibres pass through a further graphite guide block with 20 holes in it, disposed immediately in front of a bank of 20 axial jet oxy-hydrogen blowpipes, which entrain the fibres and blow them forward in the form of wool.

The wool is finally collected on a rotating drum about 3 ft. diameter, with wire meshing on the periphery. The collecting drum has its speeds adjusted so that no tension is created on the forming wool. This machine is mainly destroyed, and therefore could not be seen in operation, but the method has been previously mentioned in C.I.O.S. Report No. XXVIII - 67. Item No. 21, and B.P. 507951.

NON-TRANSPARENT FUSED SILICA.

(1) "Rotosil".

Tubes or pipes are formed in a steel rotating furnace tube, using Dörentrupe Silica Sand (approx. 99.7% SiO_2) obtained from Lippe, the heating being supplied by an opposed electric arc using current at 220 volts.

The sand is preformed to the required annular depth in the furnace tube by sweeping the bore with a forming tube, controlled externally from brackets mounted on the electrode holders.

The speed of rotation of the furnace tube is varied according to the size used and is arranged to give a common peripheral speed of about 400 ft/min.

The electrodes consist of graphite ends coned into copper electrode tubes which are arranged for water cooling; these in turn held in adjustable clamps carried from overhead carriages designed to run along steel structure runways disposed axially at each end of the furnace. The arc is struck from one end and progressively fed throughout the length of the furnace at a controlled speed to give the necessary conditions of heating to form the desired wall thickness. A minimum of 20 mm. of sand is left unfused, acting as a heat barrier.

Heavy walls up to about 2" can be formed on relatively small tubes, whilst with the maximum diameter made, namely: 350 mm. (about 14") external, the maximum permissible wall to avoid cracking has been found to be 15 mm. Small sized tubes can be ground on the surface and re-drawn in the furnace previously described, into satin surface tubing.

Building up of tube assemblies and special apparatus is carried out with blowpipes, whilst a limited grinding department deals with the grinding of Rotosil tubes etc.

(2) Moulded Articles

Crucibles up to 15" diameter have been made, but their present small plant can only produce articles about 5" external diameter x 3½" deep, and they are manufacturing such basins with a wall thickness of about 3/16". Experimental tea cups had been made, but the costs were too high to be commercial.

The furnace consists of a turntable carrying 3 rotating tables, each driven direct from a motor. The tables are filled with sand, pre-shaped by hand formers and scoops, and at the fusing position, an arc using 3 electrodes is slowly lowered, and progressively sinters and fuses the shape formed.

A considerable time will elapse before they can attain their pre-war production levels, as apart from their plant dislocation, their buildings are badly damaged.

6. SCHÖNWALD PORZELLAN FABRIK.

This factory is located at Schönwald, 2 km. from Selb, North-Eastern Bavaria. It belongs to the Kalla group as does the Hescho concern, the latter being located in the Russian zone of occupation. A visit was paid on the 19th October, 1945, in an endeavour to contact some of the Hescho technicians and obtain details of various ceramic turbine blades and stator assemblies which the Hescho firm had made.

The former technical director of the Schönwald Porzellan Fabrik, Dr. Sauer, was interviewed but he had no knowledge of the whereabouts of any of the technical staff of Hescho nor of any of their processes and compositions.

In the Schönwald factory only household porcelain is made. During the war they did, however, apply silver films to electrical porcelain for Hescho who sent the parts to the Schönwald factory for this process. The silvered parts were then shipped back to the plant in Thuringia. This part of the plant was inspected.

The silver solution, which appeared to be a silver colloid in an essential oil medium, and supplied by Degussa, Frankfurt/Main (Reference Nos. 103 and 110), is sprayed onto the ceramic through a mask to give the desired outline, dried in a small oven, then fired at 850°C. for two hours in a small resistance heated tunnel kiln approximately 8 ft. long. This kiln has an endless ceramic belt 6 in. wide, on which the articles passed through the hot zone. A second sprayed coat is applied over the first after firing, and the article refired. The process gives a very adherent bright silver film on the porcelain.

7. BERLIN STATE PORCELAIN WORKS, SELB.

(a) DATE OF VISIT.

This works was visited on the 19th October, 1945 and Dr. Frank, (Technical Director) and Dr. Koenig (Chief Research Chemist) were interviewed.

The Berlin State Porcelain factory was evacuated to the Paul Müller porcelain works, Selb, Bavaria, after very heavy damage to their factory in Berlin in a raid on 22nd to 23rd November, 1943.

(b) CERAMIC TURBINE BLADES

The evacuated factory had made turbine blades for the Hermann Goering L.F.A. which were promising from the aspect of resistance to thermal shock. Enquiry revealed that the work on these had been done by a Dr. Müller whose whereabouts were unknown but believed to be in custody as an S.S. suspect.

Personnel questioned said they had no knowledge of the processes or compositions in making the blades, but later admitted they were restarting experimental work on the same kind of material having the previous day received an order from the Hermann Goering L.F.A. (now controlled by the R.A.F.) for a new supply of silicon carbide blades. They were proposing to use a mix containing 70 per cent silicon carbide passing a sieve having 80 apertures per sq.cm. and resting on a sieve having 250 apertures per sq. cm. and 30 per cent of Zettlitz kaolin. Shaping was to be done in the plastic condition in plaster of paris moulds. After firing at 1400° to 1500°C. a porcelain glaze to which a proportion of alumina was added was to be applied and the products refired at the same temperature. A simple porcelain glaze does not wet silicon carbide.

(c) BERLIN PORCELAIN COMPOSITIONS.

Some details of the Berlin porcelains were obtained. The first four are claimed to be impermeable to gases. The remainder require to be glazed with a felspathic glaze.

K Mass

The melting point is given as 1800°C. and the highest temperature of use as 1700°C.

A grog or sinter composed of 60 per cent calcined alumina and 40 per cent felspar is first prepared by grinding these together and firing for three hours at 1500°C. This grog is then ground, mixed with Zettlitz or Halle clay in equal proportions by weight, shaped and fired at 1500°C. for three hours.

Prokorund I

The melting point is given as 1900°C. and the highest temperature of use as 1800°C.

A grog is made from 50 parts calcined alumina and 2½ parts Zettlitz clay, by firing at 1400°C.

The following composition is then prepared:-

40 parts Zettlitz kaolin (raw)

50 parts Zettlitz kaolin (fired at 1400°C.)

10 parts grog.

This is ground and articles made from the mass fired to 1600° to 1700°C.

Prokorund 2.

This mixture comprises 50 per cent Zettlitz kaolin (Raw) and 50 per cent calcined alumina. The articles are fired to 1700°C.

Prokorund 3.

This mixture consists of 30 per cent Zettlitz kaolin (raw) and 70 per cent calcined alumina. The articles are fired at 1700° to 1800°C.

Marquart.

The melting point is given as 1820°C. and the highest temperature of use as 1700°C.

The mixture comprises 46½ per cent calcined alumina, 52 per cent mixed clays and 1½ per cent feldspar, and is fired at 1500°C.

D2 Mass.

The melting point is given as 1850°C. and the highest temperature of use as 1700°C.

The mixture comprises 50 per cent fused alumina (passing a mesh of 150 openings per sq. cm.) and 50 per cent Zettlitz kaolin and is fired at 1500°C.

D3a Mass.

The melting point is given as 1850°C. and the highest temperature of use as 1700°C.

The mixture comprises 60 per cent. fused alumina and 40 per cent. Zettlitz kaolin and is fired at 1500°C.

D4 Mass.

The melting point is given as 1920°C. and the highest temperature of use as 1800°C.

The mixture comprises 50 per cent calcined alumina (specially low in iron), 50 per cent Zettlitz kaolin and is fired at 1500°C.

(d) INSPECTION OF PLANT.

The plant was that of an ordinary porcelain factory. No special equipment apart from a horizontal extrusion apparatus for extruding porcelain pipes in large diameters (up to 25 cm.) was seen.

The kilns were intermittent and fired with coal and typical of those used for the manufacture of porcelain on the Continent. The goods for glost firing were set in the lower portion of the Kaolin at ground level and those for biscuit firing at higher levels reached from the first and second floors of the factory. No equipment for firing at temperatures higher than the 1500°C. reached in these kilns was available at the Selb factory.

(e) CONCLUSIONS.

The details of manufacture of turbine blade materials which we hoped to obtain were not available owing to the absence of Dr. Müller. Although some plant had been evacuated from Berlin there was very little specialised equipment pertaining to refractory ceramic materials available at Selb.

We gained the impression that the good quality of the products of this firm were due primarily to the care exercised during manufacture and the craftmanship of the operatives.

8. ROSENTHALL INSULATOR WORKS, SELB.

(a) DATE OF VISIT.

The works were visited on the 19th October, 1945 and the personnel interviewed were Dr. Seigler (Research Engineer), Dr. Kohl (Chief Chemist) and Dr. Ullman (in charge of resistance plant).

(b) SOME SPECIAL CERAMICS.

The above firm make technical porcelain such as high frequency low lose ceramics, electrical porcelain for close tolerance work, special fixed carbon resistors, heater ceramics, etc.

A.E.G. Berlin had asked them to make ceramic turbine blades in small quantities for experimental purposes, but none had been made before the end of the war. A few experimental mixes which it was considered might give high strength material were made and samples in rod shape had been sent to A.E.G. for test but no results were known. A composition considered the most promising had the following composition:-

25 per cent hydrated alumina.
25 per cent calcined alumina.
30 per cent kaolin (two plastic clays).
15 per cent grog (from fired mix of above 3 constituents).
5 per cent titanium dioxide.

It was fired at 1450°C.

This material was claimed to have a high compressive strength (about 12,000 kgm./cm.^{2}). Venturies had been made from it for the Hermann Goering L.F.A., Volkenrode.

They had also experimented with silicon carbide - clay mixes and found the following compositions good for resistance to thermal shock.

(a) 40 per cent silicon carbide
60 per cent high plastic clay

(b) 20 per cent silicon carbide.
20 per cent fused alumina.
60 per cent high plastic clay.

The composition of the electric porcelain for making shapes to close tolerances was given as:-

Talc,	42 per cent
Sericite,	48 " "
Plastic kaolin	6 " "
Calcium bentonite,	4 " "

The sericite mineral is of the horneblende type and approximates to the composition quartz 30 per cent, mica 70 per cent. This mix has a shrinkage of 6 per cent in the direction of pressing and 8 per cent in the direction at right angles on firing to cone 9.

(c) SPECIAL CARBON RESISTANCES

A visit was also paid to a subsidiary factory of the Rosenthal concern making small fixed carbon resistors, consisting of a film of fused glass and graphite on a ceramic former. The details of the manufacture of these were obtained.

Composition of glass used.

Pb_3O_4,	40 parts	
B_2O_3,	10 "	
Na_2CO_3,	5 "	
Silica,	15 "	(fine water ground)
Jena glass	10 "	
ZnO,	10 "	
CaO,	5 "	
Feldspar,	5 "	

This is melted in an electric furnace, granulated in water and finely ground in a pebble mill for one week. The ground glass after drying is mixed with aqueous colloidal graphite in varying proportions, according to resistance required.

<u>Example 1</u>: for 50 - 100 ohms,
1 part glass
1 part colloidal graphite
1 part water

<u>Example 2</u>: for 1,000 ohms,
5 kgm. glass
1.2 kgm. graphite
3.2 kgm. water

<u>Example 3</u>: for 3,000 - 5,000 ohms,
5 kgm. glass
1 kgm. graphite
3.2 kgm. water

The glass graphite mixture is sprayed onto the ceramic formers, hollow cylinders, a film thickness of 0.1 mm. being aimed at in all cases. Spraying is carried out on a rotary table, a hot air dryer forming part of the cycle. The parts are now fired to 800° to 900°C. in a tunnel kiln, nichrome heated, the firing being carried out as quickly as possible, the average time being 1 to 2 minutes. The glass graphite film is fused at this stage. End caps of brass are now pressed on, the whole is lacquered, then the fused film cut on a spiral cutting machine to the desired resistance. The cutting is done with small carborundum wheels cutting right through the film. They have a fully automatic machine which is set so as to stop cutting when a prearranged resistance is reached.

The tolerance is about ± 1 per cent of the stated value.

9. GOEBEL-WERKE GROSSALMERODE.

(a) DATE OF VISIT AND PERSONNEL INTERVIEWED.

This firm, before the war, made firebricks and sillimanite, corundum and silicon carbide bricks. It was inspected on the 25th October and Herr A. Schoddel the commercial manager and Herr G. Riemann, the technical manager replied to the questions put.

(b) PROCESSES.

<u>Corundum Bricks.</u>

Corundum is bought from Elaktroschmeltz Werke, Kempten or from Lonza in the required grain size. The usual mix consists of 40 per cent corundum, 0 - 3 mm. in grain size; 40 per cent corundum, 0 - 1 mm. in grain size and 20 per cent finely ground clay. The clay was supplied by Schifferer and Kiercher, Allendorf, Kreis Wetzlar. Its analysis was given as: SiO_2, 44.3 per cent; Al_2O_3 and TiO_2, 38.14 per cent; Fe_2O_3, 1.3 per cent; loss-on-ignition, 12.8 per cent; refractoriness, cone 34. Shaping is carried out by hand tamping of the mix at a water content of 4 to 5 per cent. Firing is to cone 10 (1300°C.). It was claimed that the ta value (temperature of subsidence 6 per cent from maximum height) under a load of 2 kgm. per sq.cm. is 1730°C. and the te value (temperature of 40 per cent subsidence) is 1780°C.

Other products of lower alumina content are made and in pre-war days sillimanite products were also supplied.

Silicon Carbide Bricks.

Similar procedures are adopted for the manufacture of silicon carbide shapes. The materialis purchased in the desired grades and bonded with some Allendorf clay. For a finely graded mix the proportions are given as: 42.5 per cent silicon carbide, 0 - 0.1 mm.; 42.5 per cent silicon carbide, 0 - 2 mm.; 15 per cent fine clay; 0.5 per cent addition of sodium silicate. For a coarser mix the proportions are identical except that 42.5 per cent silicon carbide, 0 - 3 mm., is used in place of the 0 - 2 mm. grade. firing is to Seger cones 10 to 11 (1310°C.).

(c) EQUIPMENT.

The main output from the works is chamotte firebricks and the equipment is therefore arranged mainly to this end. For chamotte preparation there are provided a jaw crusher, screen, and a set of rolls. The material passing the screen is conveyed to the storage hopper, the oversize passed to the rolls for further crushing. The storage silos are of metal discharging at the bottom. The clay is treated similarly. The grogged clay mix is prepared by mixing first dry in an Eirich mixer (60 parts of grog to 40 parts of clay), followed by a plastic mixer arranged as a vertical pug from which the mass is shredded as it is discharged through a vertical grid opening in the side of the barrel. The bricks are made either in hand presses or in simple plastic machine presses.

The special corundum or silicon carbide mixes are shaped by hand tamping methods after first mixing the ingredients in an Eirich mixer. Wooden moulds lined with 3/16 in. steel plates are in use. A metal tool is used for tamping and the tamped surface is roughened by scratching before each fresh addition.

The chamotte bricks are dried in chamber dryers heated by waste heat abstracted from the cooling goods in the kiln. Temperature and humidity are under control.

Firing of the ware, both firebrick and silicon carbide, is carried out in a continuous chamber kiln of the Hoffmann type. There are 22 chambers, with permanent walls. Each chamber measures approximately 15 ft. x 10 ft. x 7 ft. Firing is from above on to checkerwork adjacent to the permanent walls. A feature of this kiln is a secondary firing belt across the middle of the chamber. Here coal is fed from the roof of the kiln on to checkerwork of a more open pattern than that adjacent to the division walls. This subsidiary firing zone probably helps to achieve greater uniformity of firing.

(d) CONCLUSIONS.

This works, is probably the most important producer of refractories in the Kasell area. It appeared to be competently directed but nothing really noteworthy was encountered in manufacturing technique or equipment.

APPENDIX I.

LIST OF REPORTS ON THE MECHANICAL TESTING OF CERAMICS AT THE HERMANN GOERING LUFTFAHRTFORSCHUNGANSTALT VOLKENRODE, NEAR BRUNSWICK.

(1) Zusammenstellung der bisheringen Versuchsergebnisse 8 Dusen. May 7th, 1941, NoF 464F/41.

(2) Flichkraft Zugversuch an keramik Schaufelproben by Kalisch, September, 1943, No. 2092.

(3) Versuche zur Bestimmung der Warmefestigkeit von Keramik, 18th August, 1944, No. 860701/1. = HEC {10683 548

FD 3929/47

(4) Festigkeit keramischer Werkstoffe und Warmebelasbarkeit keramischer Bauteile by Dirksen F22/45. = HEC 5556 / 5539

(5) Versuche an Dusen aus Warmfesten Werkstoffe No. 86701/2. = HEC. 10,681

APPENDIX II

VISITS TO WORKS OF SECONDARY INTEREST.

AUG. GUNDLACH, GROSSALMERODE, BEZ KASSEL

C21/921 This firm specialises in the manufacture of Plumbago
C22/3241 Crucibles, Covers and Stands.

DATE OF VISIT

October 25th, 1945.

PERSONNEL INTERVIEWED

Mr. Miller - Proprietor. Works seen and questions answered.

GENERAL CONCLUSION

Materials and methods used, and quality of product, all indicated that no progress has been made for many years.

RAW MATERIALS

Main raw materials used were:-

Passau Graphite - 93/95% Carbon. Poor small size crucible flake.

Klingenberg Clay - Well-known crucible clay, refractoriness Cone 32/33.

Grossalmerode Clay - Well-known. Refractoriness Cone 30/31.

Small quantities of electric furnace products were also used. See comments under "Mixings".

MIXINGS

The mixings employed were of old-fashioned convential type containing 40-50% of plumbage bonded with clay and sand, with up to 15% of electric furnace products where extra resistance to slag or flux was required.

In the case of crucibles used for aluminium, as much as 40% of bond clay was used to give greater protection against perishing or oxidation of the plumbago at the lower furnace temperatures employed in melting this metal. Resistance to cracking must, in consequence, have been sacrificed.

The mixing employed in steel melting crucibles was of the familiar highly grogged type containing only 25% plumbago. The electric furnace products used included the usual silicon carbide (Lonzawerke) ferrö-alloys, etc.

PROCESSES

Mixings are dry and wet mixed in Eirich mixers, then pugged and the pug rolls cut up into lengths for maturing. The matured material is repugged and the crucible "balls" built up by the old-fashioned and laborious hand method. The crucibles are shaped from these "balls" by jolleying in plaster moulds and it was claimed that the shaped crucible had a rapid grain.

The shaped products are dried slowly - taking up to five weeks in the case of the larger sizes - in steam heated drying rooms. When dry, the warm crucibles are painted with two or three coats of pottery type glazes

They are then fired in periodic kilns of the type used for firing porcelain ware, built by Paul A.F. Schurlze of Dresden. These kilns have semi-producer type fireholes and are down-draught. The gases, after passing through the floor, are drawn up vertical flues in side walls between the fireholes and, if desired, part of the products of combustion may be short-circuited from the fireholes direct into these vertical flues.

The kiln temperature was given as 1500°C, but considered to be nearer 1350°C. In firing, care is taken to maintain as reducing an atmosphere as possible above 700°C while in both heating and cooling the ware is kept above this temperature for a minimum of time to prevent oxidation of the plumbago.

KERAMISCHE SCHLEIFSCHEIBEN FABRIK (C. KREBS AND RIEDEL) KARLSHAFEN.

(a) Date of Visit and Products Made.

21/922 This firm was visited on the 23rd October and Herr
2/3242 Krebs interviewed. As indicated by its title its chief output is grinding wheels, whetstones and similar articles, prepared by bonding silicon carbide, fused alumina and emery grain. The firm Corund Union A.G., which previously had operated in Karlshafen, had been taken over by Krebs and Riedel.

(b) Processes

The works is small and customary processes are employed. Most of the manufactured articles are bonded with a porcelainic bond, but rubber and sorel cement are used for some clases of ware. A typical ceramic-bonded mix consists of 80 per cent of grain (silicon carbide, emery, etc. of the required grain size) and 6.3 per cent of clay and 13.7 per cent of felspar. The abrasive grain is normally purchased correctly graded and is first mixed dry with the remaining ingredients in one of three Eirich mixers. Water and sulphite lye are then added and further mixing carried out. The wheels were pressed in hydraulic presses operating up to 40 atmospheres on the ram. The pressed wheels are dried on steam heated racks. Firing takes place in small intermittent round or rectangular down-draught kilns to a temperature of 1300°C. The firing cycle extended over 130 to 150 hours. Coal from the Kassel region is normally employed.

A small well equipped shop for dressing and trimming the wheels was provided. A test set for testing the bursting strength of the wheels was inspected.

Although no formally set-out screening plant was used one of the smaller items of interest was a circular sieve about 2 ft. in diameter vibrated by rotating of balance weight and traversed radially by 3 rotating brushes, the whole being suspended by a stirrup. The sieved product passed direct into paper sacks. The machine was used for checking the grading of some of the batches of grain. It was made by Fraisenet of Chemnitz.

BECKER UND PISCANTON
CROSSALMERODE, KASSEL

(a) Date of Visit and Products Made

C21/920 22/3243 This firm specializes in the manufacture of graphite crucibles, covers and stands. The works manager was away at the time of the visit on the 25th October, 1945 and we were shown round the works by a foreman.

(b) Plant and Processes.

The plant, although larger than Aug.Gundlach, was no more up to date. A ball mill is used for grinding the bond clays, some of which are local and some Bavarian (Klingenberg). Mixing and tempering are carried out in an edge runner pan of old-fashioned type with double rolls and a narrow grinding surface. The mix is then pugged and stored in a cellar for 3 weeks.

The shaping is carried out in plaster moulds, a solid ball being hand shaped first. This is allowed to dry for 4 to 5 days and then the interior is cut out with a steel faced tool. A block and tackle are used to help in the manipulation of heavy shapes. The crucibles are dried on steam heated racks. They are then glazed inside and outside and fired in the same type of Schulze kiln. Temperature of firing was given as 1200° - 1300°C.

It was clear that the quality of the product at this works was attributed more to the care and craftmanship of the workers than any excellence in the plant equipment or its technical control.

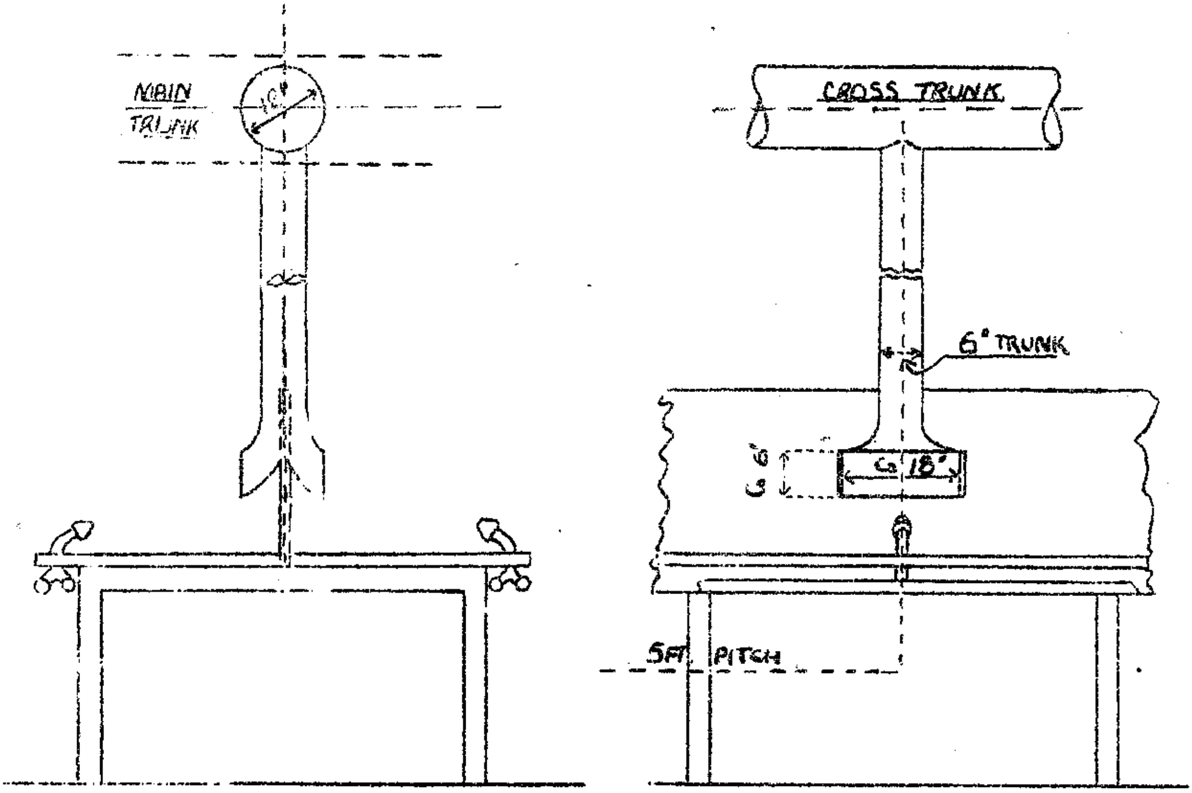
GOEBEL AND SOHN, EPTERODE,
21/916a NEAR GROSSALMERODE.

22/3240(a)

This works was visited on the 24th October and the proprietor interviewed. It proved to be a small concern making grogged fireclay crucibles and firebricks. No equipment of real interest was seen, in fact the plant was old-fashioned. The clays of the locality were used. For crucibles, the proportion of chamottes to raw clay was 3:2. A set of vibrating 2-bank screens was available for grading the grog into three sizes. A trommel was used for dealing with the coarser grog used for the larger articles. The bond clay was ground in a ball mill. The grog-clay mixture was prepared in a vertical pug mixer. Crucibles were shaped by a jolleying process on a wheel. Bricks were made by hand moulding and by wire-cutting to a limited extent. The best clay was reported to contain 38 per cent Al_2O_3 in the fired state. Crucibles were also made from a mix containing 2 parts of fireclay, 1 part of sand and 1 of used graphite crucibles. The graphite is added merely to increase porosity and thermal shock resistance. Firing is to 1100°C.

HERAEUS HANAU.

SKETCH No. 1



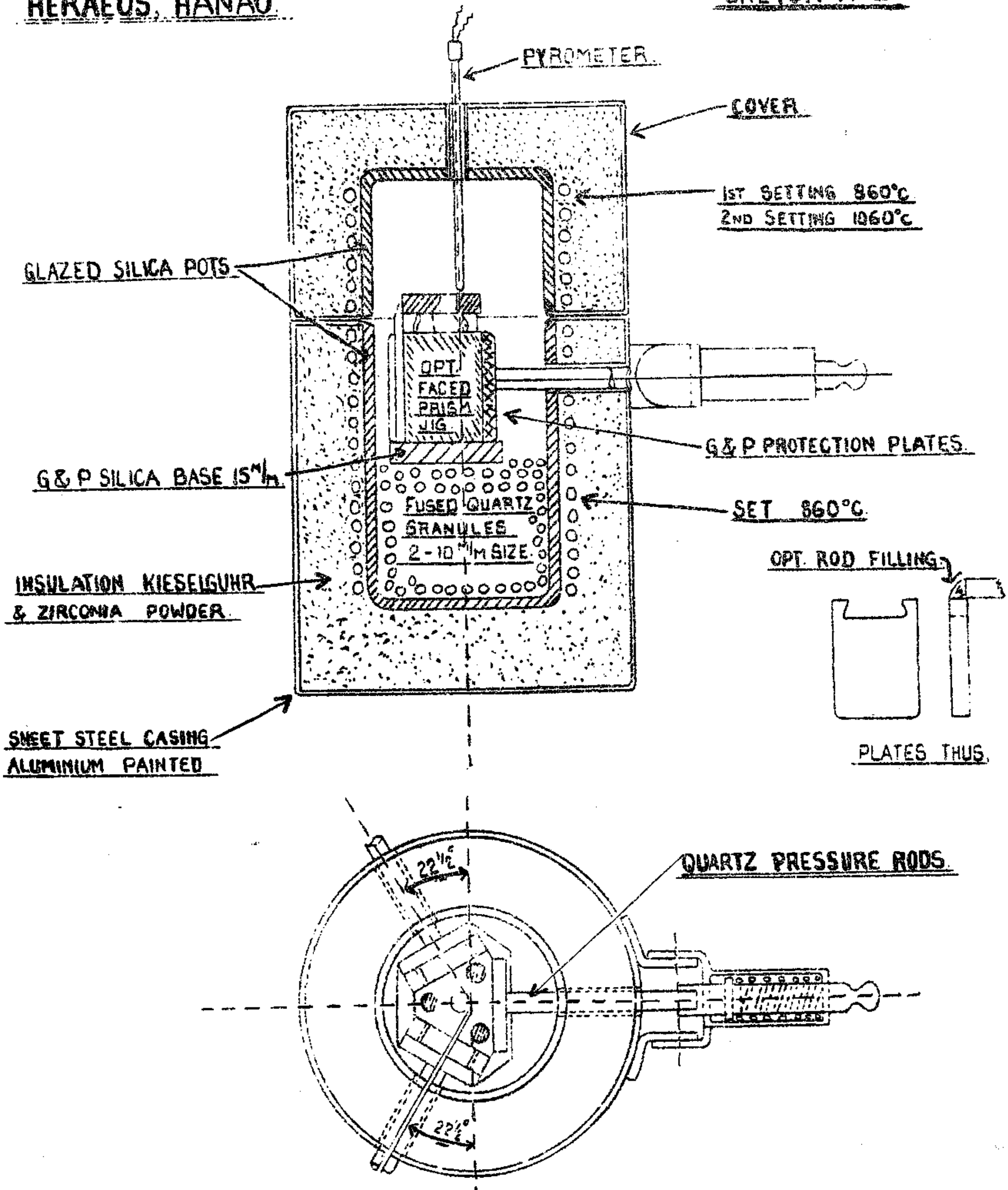
4 THUS PER ROW — 8 B.Ps.

NORMALLY OXY-HYDROGEN USED, BUT AT PRESENT FORCED TO USE OXY-ACETYLENE.

SKETCH OF BLOW-PIPE EXHAUST SYSTEM.

HERAEUS, HANAU.

SKETCH Nº2.

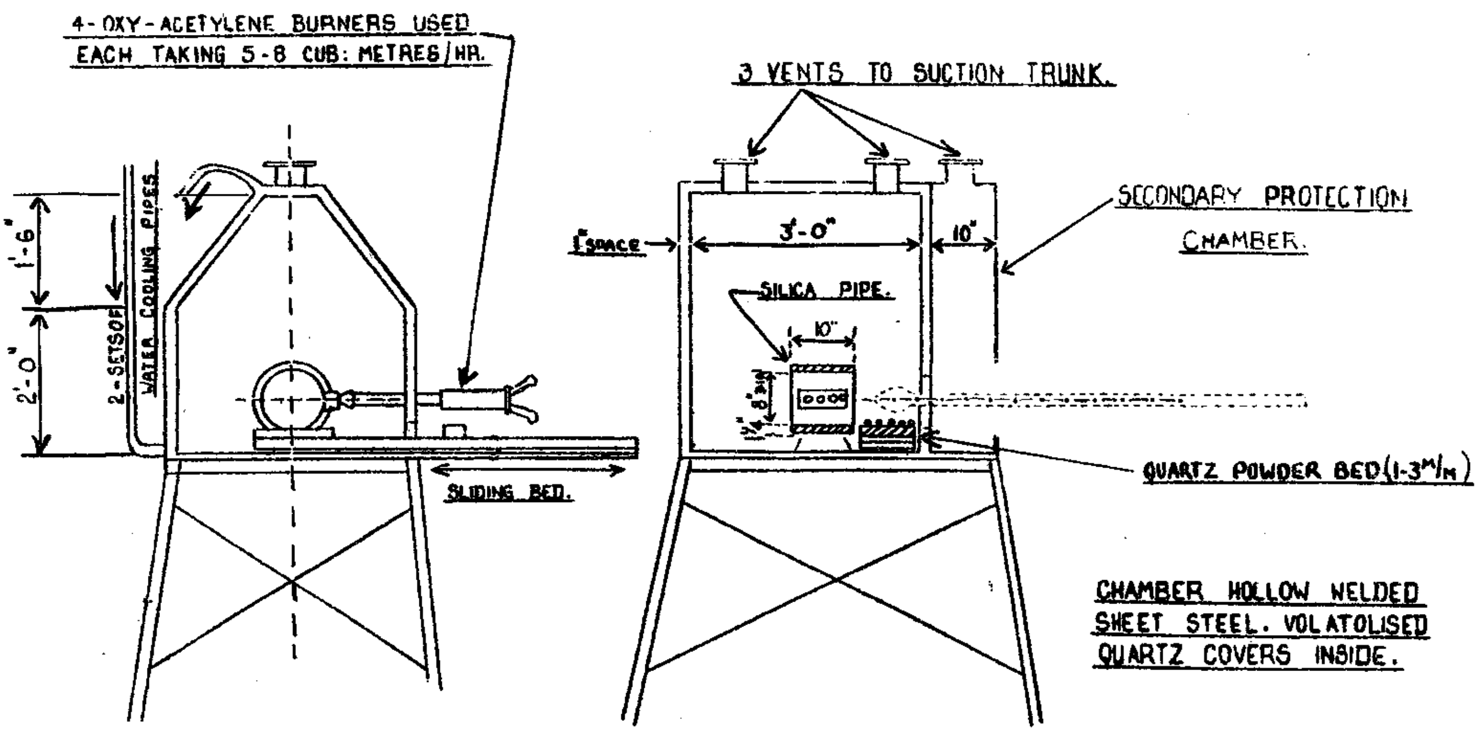


SKETCH OF SPECIAL FURNACE FOR OPTICAL QUARTZ PLATE ASSEMBLY (SCALE Ca: 3" = 1 FT.)

HERAEUS HANAU

SKETCH No. 3.

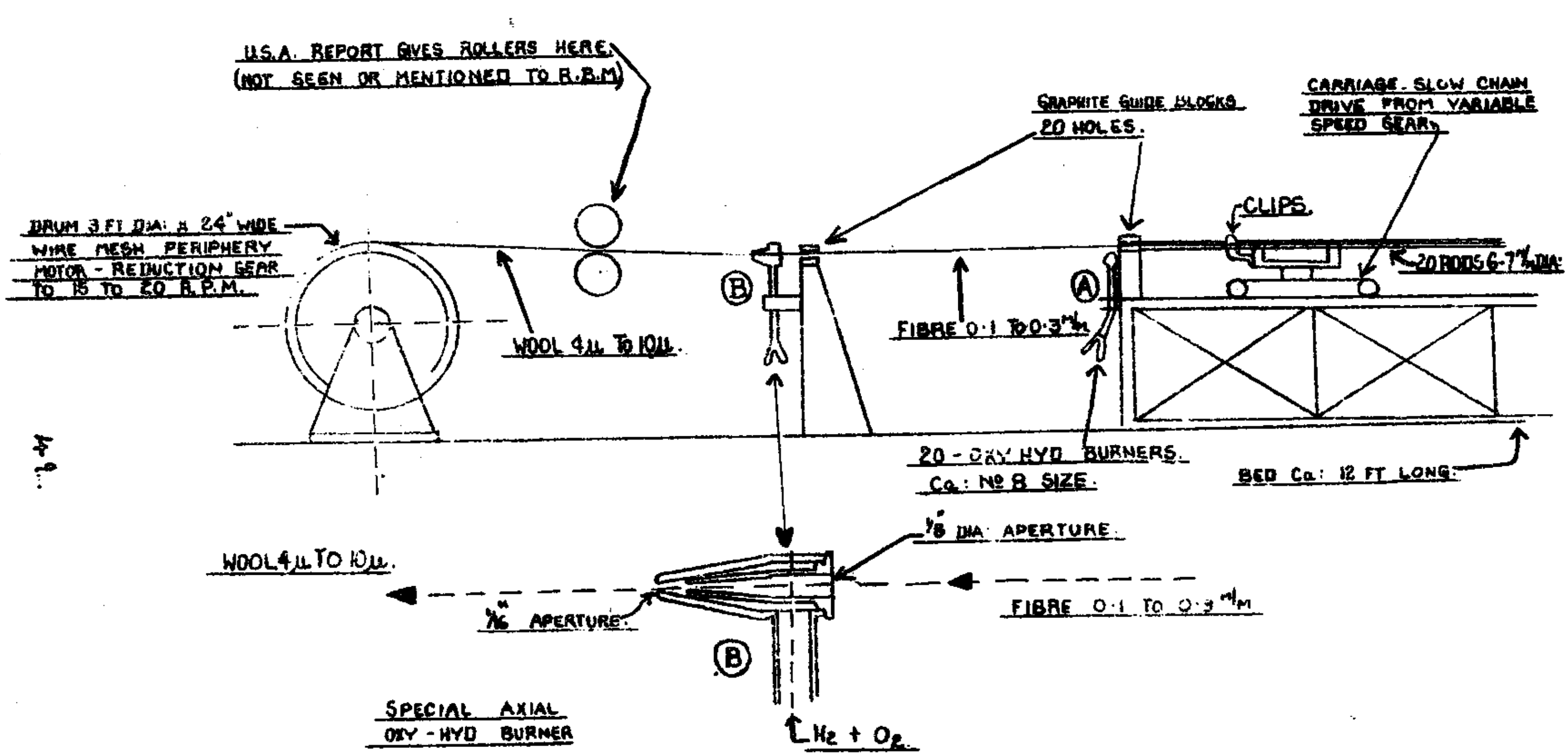
FURNACE FOR MAKING NORMAL QUALITY BILLETS



THE FUSION (QUARTZ END INITIALLY) IS HEATED, PULLED BACK, ROLLED ON POWDER BED, & RE-FUSED PROGRESSIVELY, THE HOLE IS KEPT OPEN BY PUSHING ON TO GRAPHITE TAPEP PIN. IF FUSION PROPORTION TO BE MODIFIED, IT IS HEATED UP, BLOWN UP, REHEATED & PULLED DOWN AS REQUIRED.

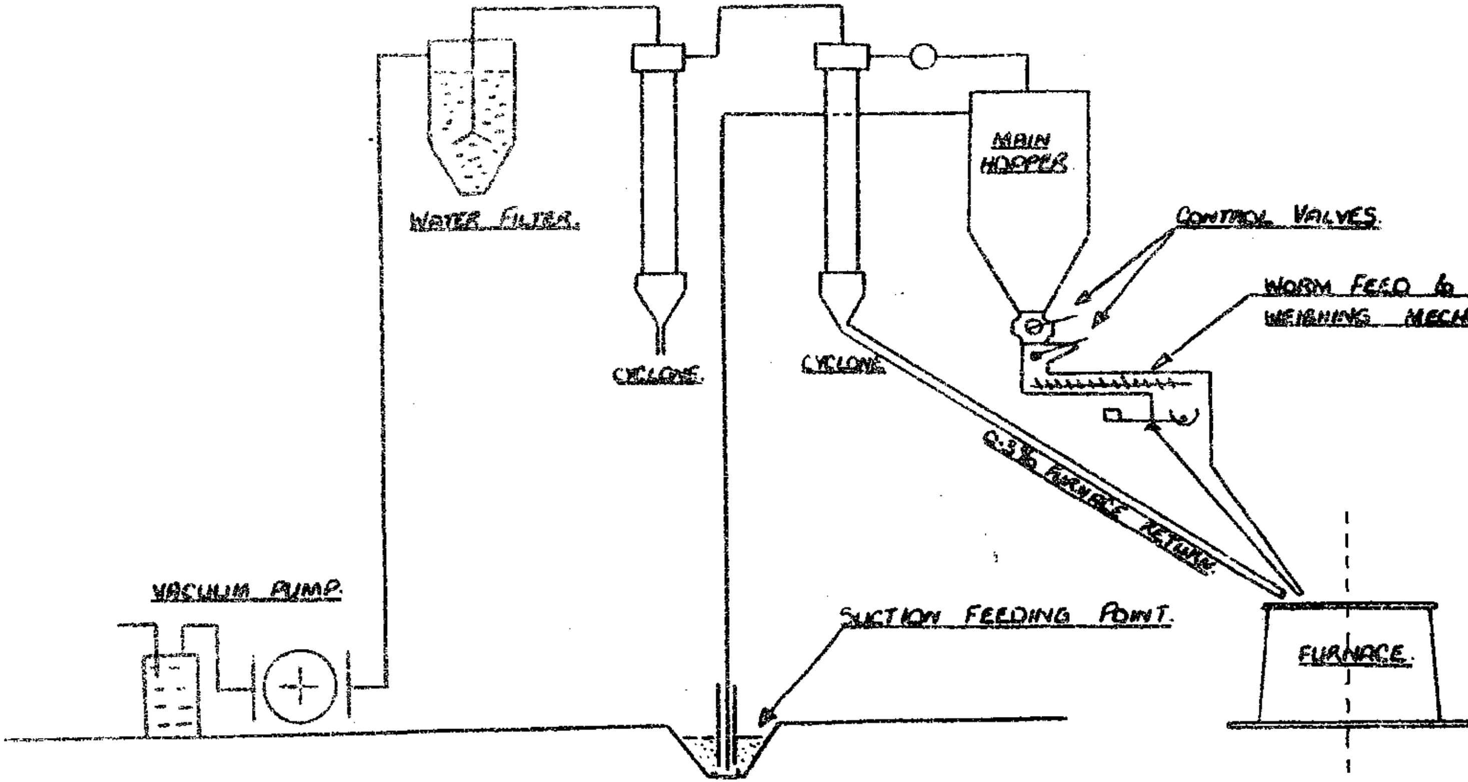
HERAEUS, HANALL

SK: Nº 4.

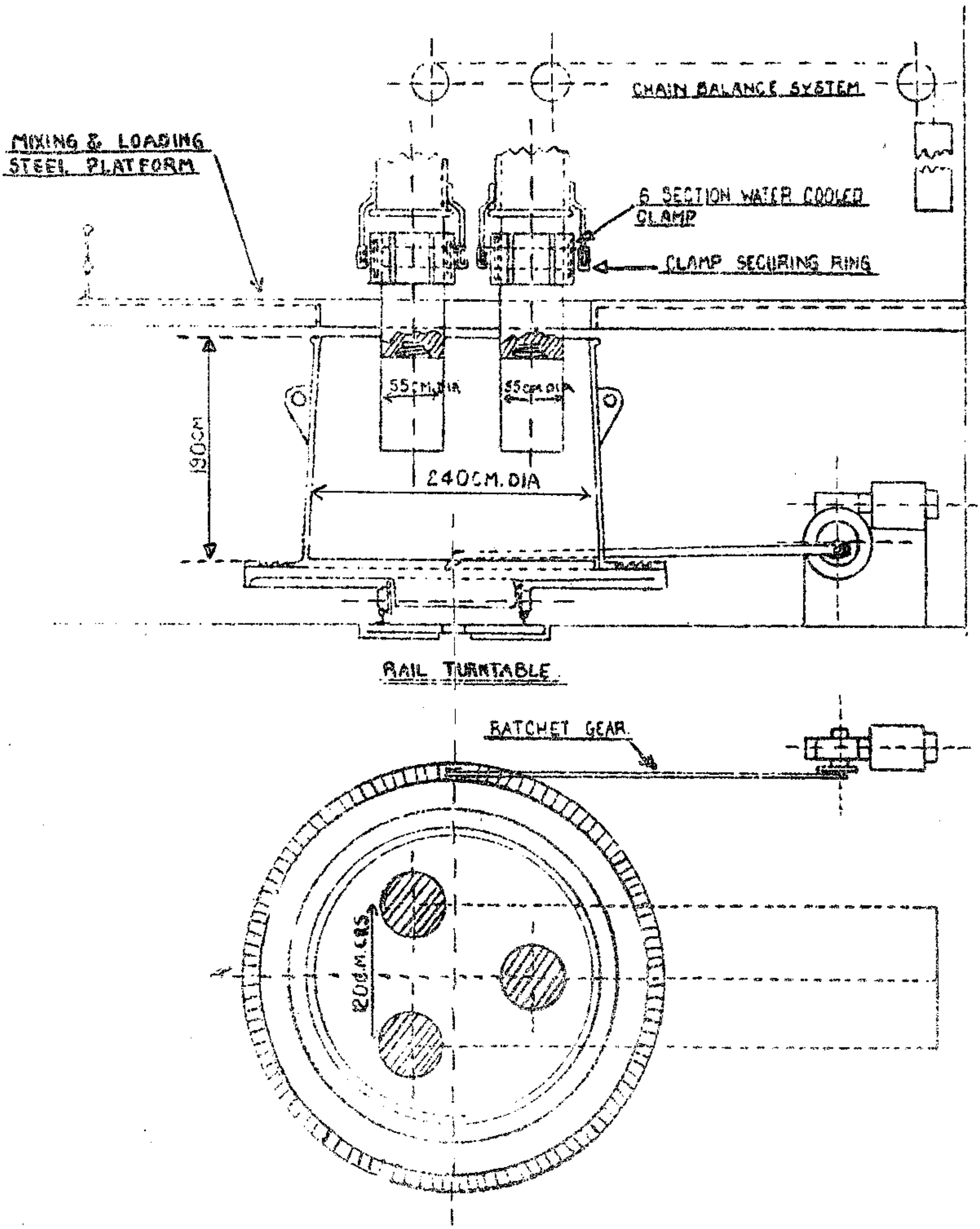


49...

FUSED ALUMINA (WHITE) FURNACE.



DIAGRAMMATIC LAYOUT OF FEEDING SYSTEM.

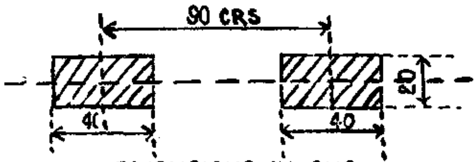


LAYOUT OF FUSED ALUMINA (BLACK) FURNACE

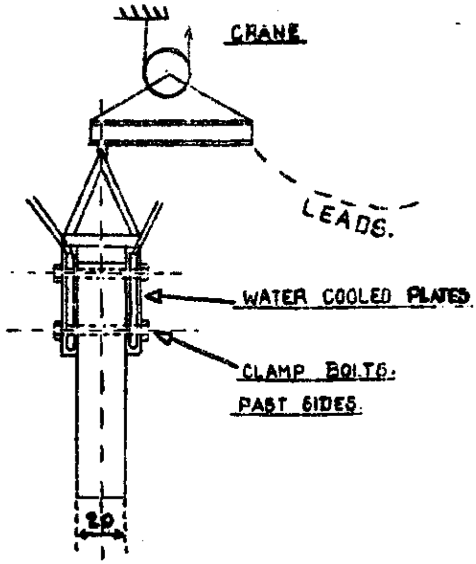
FELDMÜHLE

SKETCH Nº 3.

FUSED ALUMINA (WHITE) FURNACE
ELELTRODE ARRANGEMENT



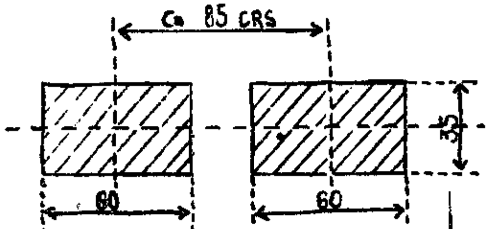
DIMENSIONS IN CMS.

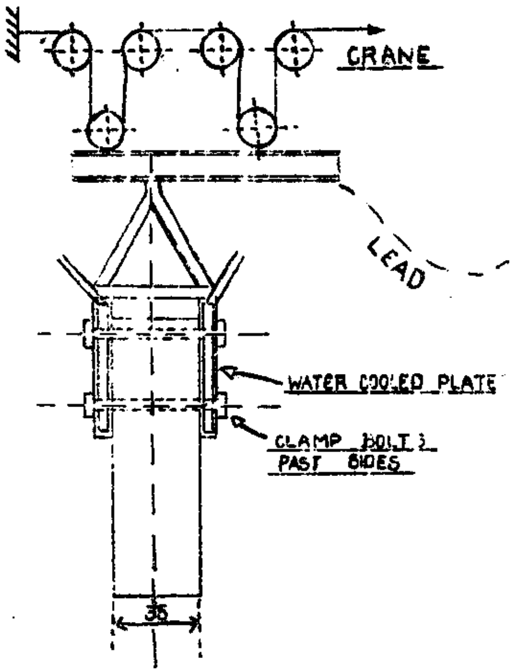


FELDMÜHLE.

SKETCH Nº 4

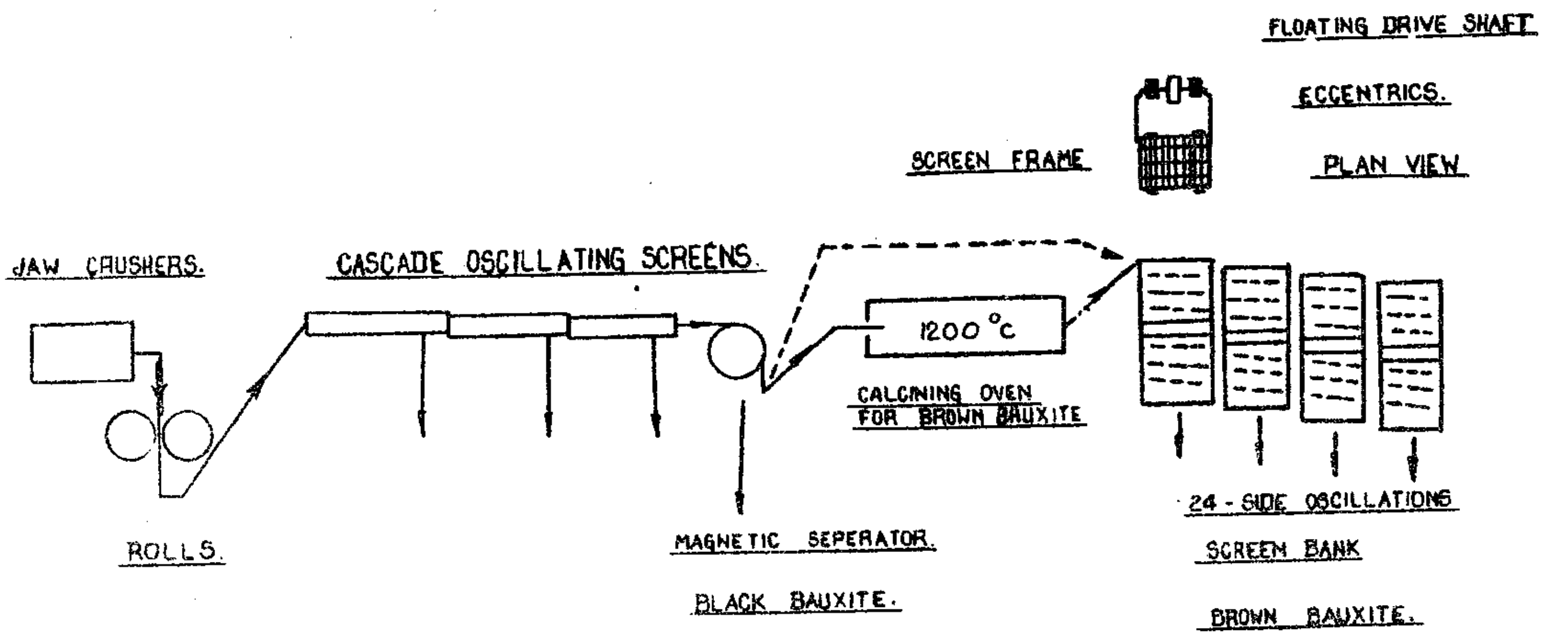
FUSED MAGNESIA FURNACE
ELECTRODE ARRANGEMENTS





FELD MÜHLE.

SK: № 5.



DIAGRAMMATIC LAYOUT OF FUSED ALUMINA (BROWN & BLACK) PROCESSING.