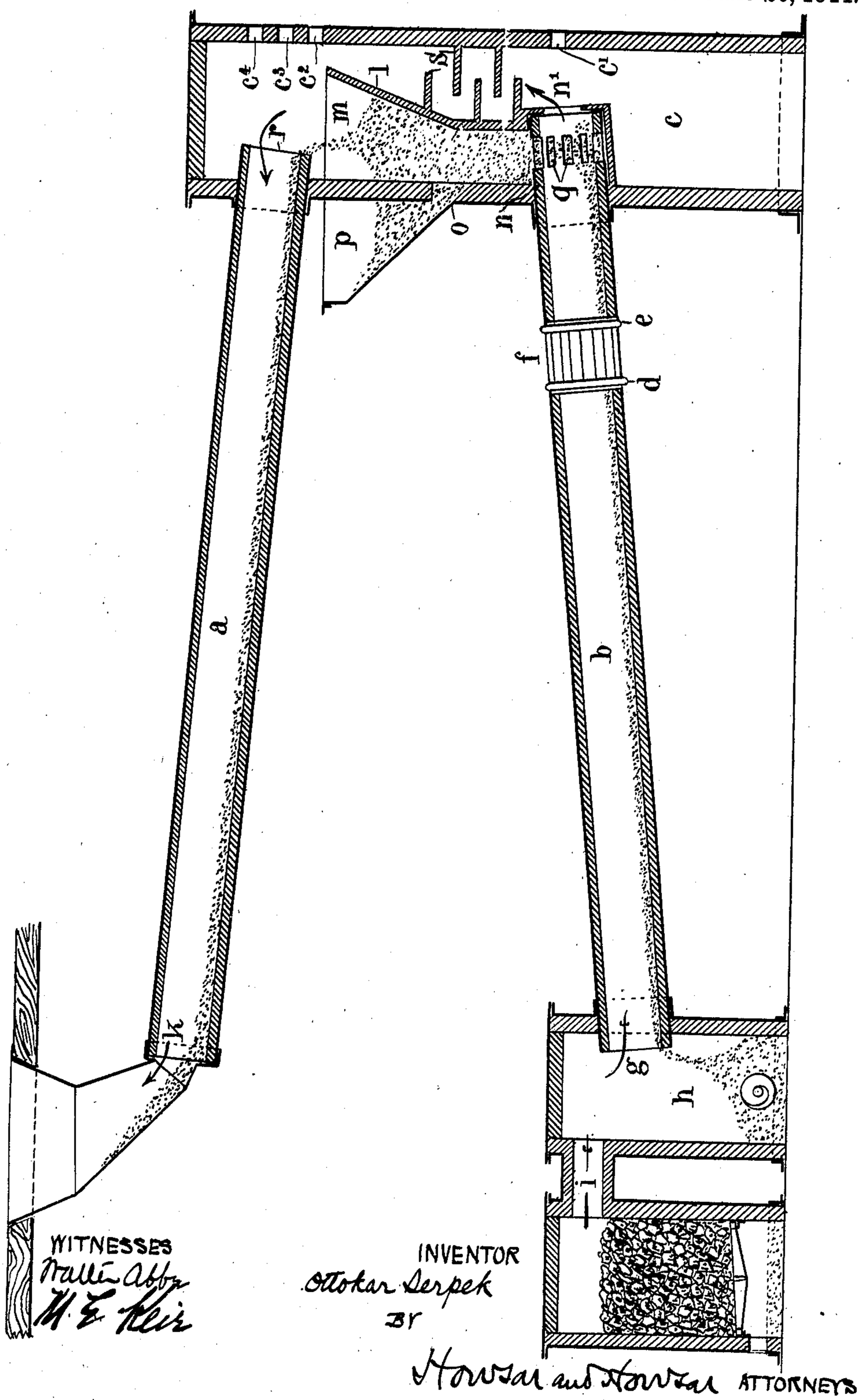
O. SERPEK.

PROCESS FOR THE MANUFACTURE OF ALUMINIUM NITRID.

APPLICATION FILED JUNE 21, 1910.

996,032. Patented June 20, 1911.



UNITED STATES PATENT OFFICE.

OTTOKAR SERPEK, OF PARIS, FRANCE, ASSIGNOR TO SOCIÉTÉ GENERALE DES NITRURES, OF PARIS, FRANCE.

PROCESS FOR THE MANUFACTURE OF ALUMINIUM NITRID.

996,032. Specification of Letters Patent. **Patented June 20, 1911.**

Application filed June 21, 1910. Serial No. 568,159.

To all whom it may concern:

Be it known that I, OTTOKAR SERPEK, a subject of the Emperor of Austria-Hungary, and a resident of 12 Rue Roquepine, Paris, France, have invented a new and useful Process for the Manufacture of Aluminium Nitrid, of which the following is a specification.

The present invention relates to an apparatus for the manufacture of aluminium nitrid, by the method which consists in the fixation of nitrogen by alumina bauxite, or other aluminium ores in presence of carbon at a high temperature.

The object of the invention is to obtain aluminium nitrid practically free from carbids or silicon compounds and at the same time to reduce to a minimum the quantity of heat expended. This object is attained by carrying out the reaction properly so-called by electrical heating whereas the preliminary heating of the substances is effected by the combustion of the carbon monoxid which is developed in the reaction and of that which is formed by the gas-generator supplying the necessary nitrogen, while the preliminary heating of the gases takes place by the cooling of the nitrid as it leaves the electric furnace.

The principle of the apparatus is hereafter described with reference to the annexed diagrammatic drawing.

The apparatus employed for the manufacture consists of two revolving cylinders *a b* similar to those used in the cement industry. These two cylinders both enter at one extremity a fixed chamber *c*. The upper cylinder *a* is a bauxite-calciner, heated by gas as hereafter described. The lower cylinder *b* is similar to the preceding except that in the part *d e* there is inserted an electrical resistance-furnace *f*, preferably of the type described in my application for Patent No. 568158 filed June 21, 1910. This furnace is made detachable so that it is readily replaced, for there is considerable wear upon the resistances and the furnace has to be renewed long before the tubes *a b* are worn out. The extremity *g* of the cylinder *b* leads to a fixed chamber *h* which also receives through a passage *i* the gases from a retort or generator *j* working under pressure.

The extremity *k* of the cylinder *a* is arranged to serve for the removal of the gases burned in this cylinder and for the supplying of the bauxite.

In the chamber *c* a partition *l* forms with the walls of this chamber a hopper *m* of which the contents press upon the head *n* of the cylinder *b*. This hopper receives the bauxite which has been calcined in the upper cylinder *a*, and it also receives through the opening *o* powdered coke stored in the hopper *p*. The head *n* is provided with an opening *n'* serving for the escape of the heated gases coming from the cylinder *b*, and with openings *q* of suitable number and dimensions for the passage of the contents of the hopper *m* into this cylinder.

The operation is as follows: Ground bauxite is introduced through the opening *k* into the upper part of the cylinder *a*, and descends slowly along this tube by reason of the slope and the rotary motion; it falls at length through the opening *r* into the compartment *m*. In this compartment it mixes with carbon from the hopper *p*, and the mixture passes through the openings *q* into the lower cylinder *b*. The rotation of this cylinder, while conveying the mixture to the electrical part of the furnace where the reaction proper is to take place, acts to mix the bauxite and carbon intimately together. In the section *d e* which consists as stated of an electrical resistance-furnace, the reaction takes place which gives rise to the nitrid. The nitrogen necessary to the reaction is furnished by the gas from the generator *j*, and this gas is itself greatly enriched in carbon monoxid by the reaction itself. This enriched gas leaves by the opening *n'* passing into the chamber *c* and taking with it in the form of more or less complex compounds the greater part of the silica of the heated material. In the chamber *c* there may be produced by a suitable blast of air as at *c'* a partial combustion of the gas resulting in the oxidation of these compounds and their deposit in the form of silica. Baffling partitions *s* may be arranged to facilitate this deposit. At the top of the chamber *c* the gases pass through the opening *r* into the cylinder *a*, after having received by more forced air inlets c^2, c^3, c^4 the oxygen necessary to their complete combustion in this upper part of the chamber and in the cylinder *a* itself. This combustion supplies the heat necessary to the cal-

cination of the bauxite in the cylinder *a*. If it be desired to increase the temperature in the upper tube, combustible gases or powdered fuel may be introduced at c^2, c^3 at the same time as the air. Instead of supplying bauxite alone to this tube, the mixture of bauxite and carbon may be supplied; in this latter case the hopper *p* serves merely for the addition of the carbon necessary to compensate for the combustion of carbon in the upper tube during the calcination.

As regards the temperature employed and the methodical recovery of the heat, the following results are obtained: The mixture of nitrogen and carbon monoxid leaves the generator *j* already at a high temperature (about 400° C.) and is further heated while ascending the tube *b* in contact with the nitrid which leaves the electric furnace at temperatures of 1800°; when it escapes to the chamber *c* it is about 200°. The gases are more effectively heated for the reason that the rotation of the furnace causes a constant stirring into contact with the gases of the solid substances which are at a high temperature. In the electric furnace, the mixture of carbon and bauxite is already heated on arrival, and the nitrogen acting upon it produces aluminium nitrid. The rotation of the furnace causes a stirring of the material, and at the same time the solid particles in falling constantly in an atmosphere containing nitrogen offer to this latter a larger and constantly renewed surface for attack. Upon leaving the furnace the hot gases are in contact with the solid substances and as a result they strongly heat the latter. At the top of the chamber *c* the gases encounter an air inlet, which by reason of their very high temperature (about 1500° C.) permits the combustion of the carbon monoxid. Consequently their temperature is again further raised and as they pass through the tube or cylinder *a* in the contrary direction to the bauxite they effect the calcination of the latter. In the same way as in the lower part of the tube *b*, the stirring caused by the rotation favors the exchange of heat between the gases and the bauxite which attains a temperature of 1400° C. when it falls at *m*, while the gases leave the top of the tube *a* at about 300° centigrade.

This arrangement therefore affords a number of advantages. (1) A methodical heating of the substances takes place with as complete recovery of the heat as possible. (2) For this heating there is utilized the combustion of combustible gases produced in the generator and in the electric furnace; the quantity of heat to be furnished by the electrical energy is thereby reduced to a minimum. (3) The solid substances are mixed in an intimate manner before entering the electrical furnace. (4) An intimate contact of all the particles with the nitrogenous atmosphere in the furnace is insured. (5) The silicon contained in the bauxite and volatilized in the electrical furnace is separated by combustion and by the deposit in the chamber *c* of the silica so formed. (6) It becomes possible to calcine the bauxite before mixing it with carbon, and so to avoid any loss of carbon during the calcination.

The invention is not strictly limited to the arrangement indicated, which may be modified as practice may show to be necessary; in particular the arrangement for feeding the carbon and bauxite mixture to the lower tube and the arrangement allowing the combustion and the separation of the silicon are given merely by way of example. In the same way this apparatus may be employed for the manufacture of other metallic nitrids from oxids capable of being treated under similar conditions, with suitable modifications as required.

Claims:

1. An apparatus for the manufacture of aluminium nitrid comprising a stationary vertical chamber, a revolving inclined cylinder opening at its lower end into the upper portion of said chamber, a second inclined revolving cylinder opening at its upper end into said chamber at a lower level than said first cylinder, means for feeding ore from said upper to said lower cylinder, in combination with an electric resistance furnace arranged in said lower cylinder and means for leading a suitable gas to said lower cylinder and furnace and thence to the upper cylinder, said intermediate chamber having ports arranged therein to admit air which affords the oxygen necessary to the combustion of said gas in the upper cylinder, substantially as described.

2. An apparatus for the manufacture of aluminium nitrid, comprising a stationary vertical chamber, revolving inclined cylinders opening into said chamber one above the other at their lower and upper ends respectively, in combination with a hopper arranged within said chamber to receive the discharge from the upper cylinder and guide it to the lower cylinder, and a hopper external to said chamber but communicating with the hopper within said chamber to permit the addition of other material to the charge entering the lower cylinder, together with an electric furnace in said lower cylinder and means for leading to the latter and thence to said chamber and upper cylinder a suitable gas, substantially as described.

3. An apparatus for the manufacture of aluminium nitrid comprising two oppositely inclined rotating cylinders communicating at one end through an intermediate chamber, said lower cylinder receiving at least a portion of its charge from said upper cylin-

der, in combination with an electric resistance furnace rigidly mounted in said lower cylinder and revolving therewith, together with means for leading a suitable gas to said lower cylinder and furnace and thence to the upper cylinder, substantially as described.

4. An apparatus for the manufacture of aluminium nitrid, comprising two oppositely inclined rotating cylinders communicating at one end through an intermediate chamber, said lower cylinder receiving at least a portion of its charge from said upper cylinder, in combination with an electric furnace in said lower cylinder and means for leading a suitable reaction gas to said lower cylinder and furnace, together with a baffled passage for said gas from said lower cylinder to the upper cylinder and a port for the admission of air to mingle with said gas before reaching the baffles in said passage, for the purpose specified.

5. An apparatus of the character described comprising as an element thereof a rotary conveying cylinder carrying an electric resistance furnace through which the material treated is passed.

6. An apparatus for the purpose described comprising a calcining cylinder, a reaction furnace and means for leading the discharge from said calcining cylinder to said furnace, in combination with means for leading a reaction gas through said furnace to said calcining cylinder, together with an air intake interposed between said furnace and calcining cylinder for supporting the combustion of said gases in the calcining cylinder.

7. An apparatus for the purpose described, comprising a calcining cylinder, a mixing hopper into which said calcining cylinder discharges, a reaction furnace and means for leading the material from said hopper thereto, in combination with means for leading a reaction gas through said furnace and past said hopper to the calcining cylinder, together with an air intake interposed between said furnace and cylinder for supporting the combustion of said gas in the calcining cylinder, substantially as described.

8. An apparatus of the character described, comprising as an element thereof a rotary conveying cylinder with an electric resistance furnace detachably mounted therein intermediate its ends, substantially as described.

In testimony whereof I have signed my name to this specification, in the presence of two subscribing witnesses.

OTTOKAR SERPEK.

Witnesses:
CHARLES DONY,
JAQUES LIGNEY.