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PROCESS OF PRODUCING COMPOUNDS OF ALUMINUM, CARBON, AND NITROGEN.

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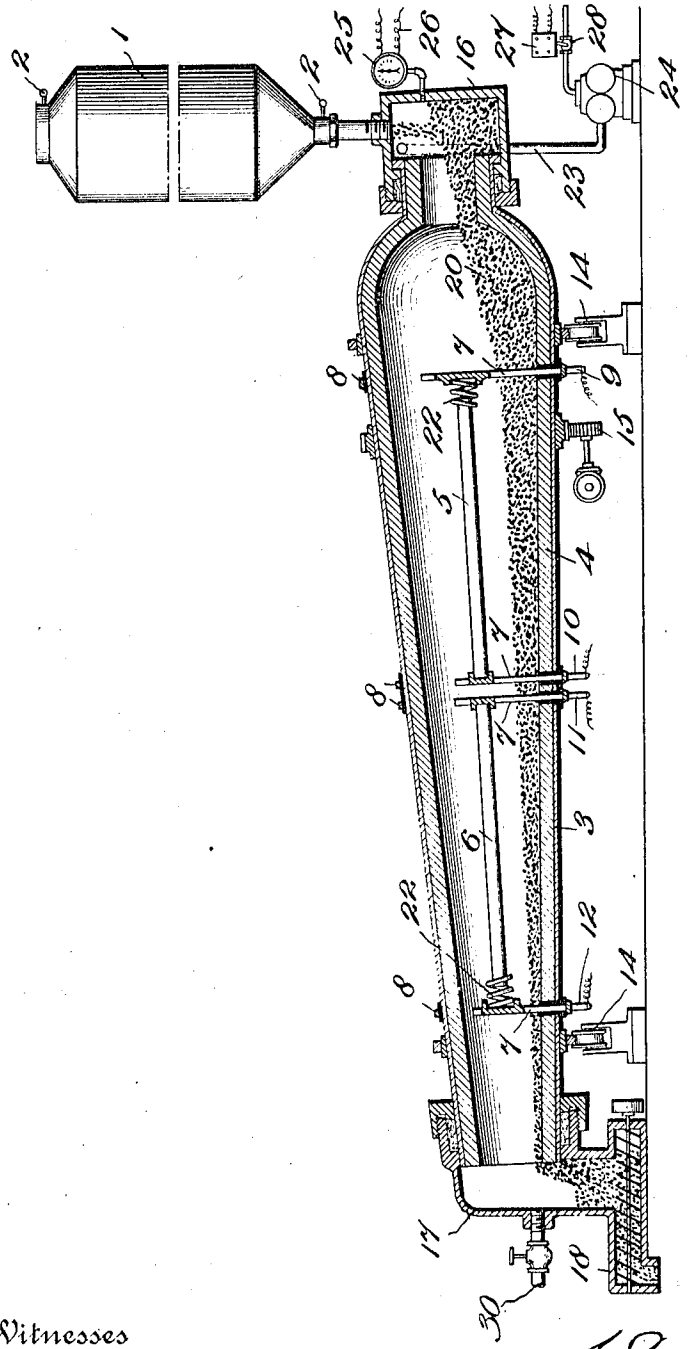
1,035,727. Patented Aug. 13, 1912.



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UNITED STATES PATENT OFFICE.

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PROCESS OF PRODUCING COMPOUNDS OF ALUMINUM, CARBON, AND NITROGEN.

1,035,727. Specification of Letters Patent. Patented Aug. 13, 1912.

Application filed September 23, 1911. Serial No. 650,973.

To all whom it may concern:

Be it known that I, SAMUEL PEACOCK, a citizen of the United States, residing at Chicago, in the county of Cook and State of Illinois, have invented certain new and useful Improvements in Processes of Producing Compounds of Aluminum, Carbon, and Nitrogen; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

This invention relates to the production of a nitrid having the formula $Al_2C_3N_6$, and has for its object to provide a process for making this product in an efficient and expeditious manner.

To these ends the invention consists in the novel steps constituting my invention which is more fully hereinafter disclosed and particularly pointed out in the claims.

Referring to the accompanying drawings forming a part of this specification, the figure is a diagrammatic sectional illustration of a furnace suitable for carrying out my invention.

1 indicates any suitable supply for the mixed carbon and alumina, 2 valves controlling the same, 3 a furnace provided with a refractory lining 4, 5 a resistor of graphite or other suitable material, 6 a second resistor, 7 supports for said resistor, 8 rings on the outside of the furnace connected with said supports 7 and adapted to make contact with the brushes 9, 10, 11 and 12 included in suitable electric circuits not shown.

The furnace is supported by any suitable means as for example, the rollers 14, and is revolved as by the means 15. 16 represents an air-tight connection between the receptacle 1 and the larger end of the furnace, while 17 represents a similar connection between the exit 18 and the smaller end of the furnace. The shape of the furnace is preferably conical with its lower side horizontal, and when rotated it therefore carries the material 20 in small particles upwardly on its inner side, and drops the same down at a point nearer the smaller end than it was before. In this way, the material is continuously fed through and evenly heated in the furnace as it rotates.

In order to allow for the expansion and contraction of the resistors, I preferably provide spring supports 22 which may be suitably made of carbon or other refractory conducting substance.

23 represents a suction pipe for the pump 24, which communicates with the interior of the connection 16 as shown, and 25 represents a suitable vacuum gage, which is provided with contacts, not shown, connecting with the wires 26.

27 represents any suitable electro-magnetic device adapted to be operated from the gage 25 and controlling the exit valve 28 of the pump, although my process may be carried out at atmospheric pressure, or even above, if desired.

Since the above apparatus forms no part of my present invention, I do not deem it necessary to disclose more of its details, and only refer to it here in order to make clearer the process now to be described.

Briefly stated, this process consists in taking advantage of the relation which exists between the equilibrium temperature and pressure of the formation of the nitrid, $Al_2C_3N_6$, and preferably in causing said nitrid to form at a temperature in the neighborhood of 1500° centigrade or lower, while the internal pressure of the furnace is below that of the atmosphere, or at say 500 millimeters of mercury or lower.

In carrying out my process, I take finely divided and dry alumina and finely divided dry carbon, preferably coke or charcoal, in the ratio of 102 parts by weight of the former to 72 parts of the latter, thoroughly mix the same, and feed the mixture into the furnace as illustrated. I find in practice that the process is facilitated if an excess of carbon over that required to satisfy the equation below, and sometimes as high at 25 or 30 per cent. is used, since it prevents the formation of aluminum nitrids such as Al_2N_2, which contains a much lower percentage of nitrogen than do the nitrids of my process. Said excess of carbon also insures the more nearly complete conversion of the alumina to the form distinguished by the formula $Al_2C_3N_6$, said excess of carbon not being injurious to the process. After the mixed alumina and carbon is in the furnace, I preferably subject it under the conditions above mentioned, by means of the

resistor 5 to a temperature of about 1500° C., which effects the formation of the nitrid in accordance with the following equation:—

$$Al_2O_3+6C+3N_2=Al_2C_3N_6+3CO.$$

As the nitrid, however, is formed and the material fed by the furnace under the electrode 6, less total heat units are required to keep up the reaction temperature, because the reaction lessens in velocity and less heat is absorbed. Therefore, I preferably include the resistor 6 in an independent circuit, and pass only sufficient current therethrough to supply the necessary heat in the furnace to correspond with the selected pressure of gas that is being maintained. The material is next cooled and discharged from the furnace by any suitable means.

Any carbon monoxid CO which may be liberated from the charge, will be forced forward by the nitrogen, as well as sucked forward by the pump, and therefore it will not reach the nitrid in sufficient proportions to cause any damage. The sucking forward of this carbon monoxid necessarily lessens its partial pressure, and therefore hastens the reaction. And, since the whole pressure in the furnace may be as low as 500 mm. it also necessarily results that the real partial pressure of the carbon monoxid is very low indeed, so that the reaction will proceed at a very much lower temperature than would be the case if the partial pressure was permitted to rise to the value it would have if the furnace were open to the atmosphere.

The gas connections, of course, must be sufficiently large to supply nitrogen fast enough to satisfy the reaction, and it is also an advantage to maintain as low a pressure and temperature inside the furnace as will bring about the desired reaction, because any silica or other impurities that may be present will not be fused and therefore, the walls of the furnace will not be gummed up as would be the case with higher temperatures.

It will be observed that my product $Al_2C_3N_6$ contains six parts of fixed nitrogen by weight to two parts of aluminum, whereas if I allowed the formation of aluminum nitrid, Al_2N_2, to take place, only two parts of fixed nitrogen to two parts of aluminum would be obtained.

The product of this process forms the subject of my copending application #650,972 of even date herewith.

It is obvious that those skilled in the art may vary the details of my process without departing from the spirit thereof, and therefore I do not wish to be limited to the above disclosure except as may be required by the claims.

What I claim is:—

1. The process of producing a nitrid having the formula $Al_2C_3N_6$, which consists in heating at a pressure below 500 millimeters of mercury in the presence of nitrogen alumina with sufficient carbon and to a temperature sufficient to produce said compound, substantially as described.

2. The process of producing a nitrid having the formula $Al_2C_3N_6$, which consists in heating alumina in the presence of nitrogen at pressure less than that of the atmosphere with sufficient carbon and to a temperature requisite to form said compound, substantially as described.

3. The process of producing a nitrid which consists in mixing finely divided alumina with carbon, the latter being in excess of that required to produce said nitrid; and subjecting the mixture under a pressure less than 500 millimeters of mercury in an atmosphere of nitrogen to the temperature necessary to produce said nitrid, substantially as described.

4. The process of producing a nitrid, which consists in mixing finely divided alumina with carbon, the latter being in excess of that required to produce said nitrid; and subjecting the mixture in an atmosphere of nitrogen under a pressure less than that of the atmosphere to the temperature necessary to produce said nitrid, substantially as described.

In testimony whereof, I affix my signature, in presence of two witnesses.

SAMUEL PEACOCK.

Witnesses:
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