May 21, 1968 A. FUCHS 3,384,455

METHOD OF PRODUCING NITRIDING AGENTS

Filed Nov. 22, 1965 2 Sheets-Sheet 1

Fig.1

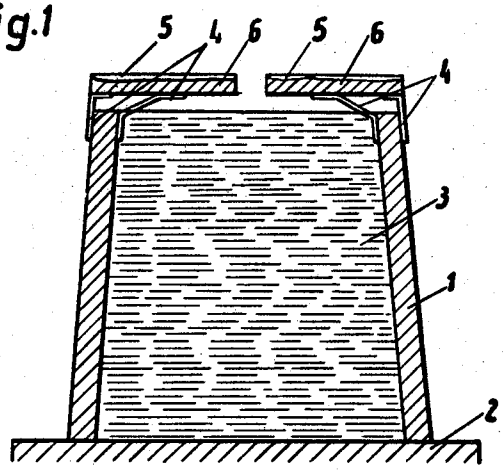
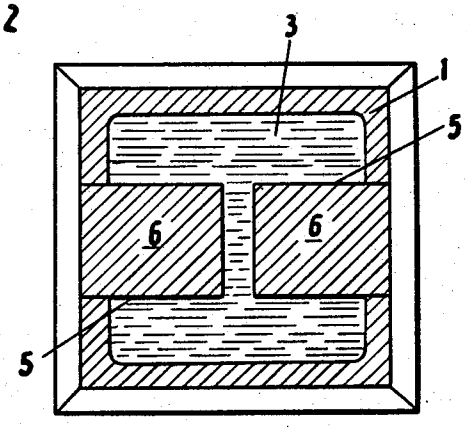


Fig.2



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May 21, 1968 A. FUCHS 3,384,455

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Filed Nov. 22, 1965 2 Sheets-Sheet 2

Fig.3

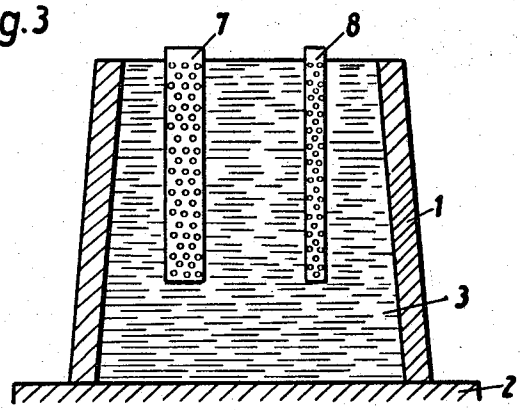
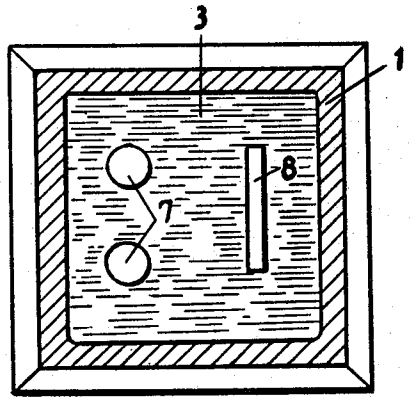


Fig.4



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METHOD OF PRODUCING NITRIDING AGENTS

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7 Claims. (Cl. 23—191)

ABSTRACT OF THE DISCLOSURE

A process for producing a nitrogen-containing metal for use as a nitriding and alloying agent which includes contacting a comminuted metal with the residual gases and the exothermic heat of reaction produced in the reaction of calcium carbide with stoichiometrically excess nitrogen to form calcium cyanamide. Such contact enriches the nitrogen content of the metal. Thereafter the resulting sintered nitrogen-containing metal is crushed to form abraded fines of the nitriding and alloying agent.

This invention relates to a method of producing nitriding and alloying agents, i.e. nitrogen-containing metals or alloys, including ferro-alloys; and particularly relates to a method whereby such agents are treated in conjunction with known processes for producing calcium cyanamide.

For nitriding and alloying steels, molten or sintered metal-containing nitriding agents are used. Molten nitrogen-containing alloy nitriding agents have the advantage of providing a high nitrogen content in the steel, despite the fact that in relation to their content of alloying elements they contain little nitrogen. For instance highly-refined FeCr contains 64 to 70% Cr but only 2 to 4% N, highly-refined FeMn contains 75 to 82% Mn and only 1.5 to 2.6% N, refined FeMn contains 1% C and only 1.8 to 2.2% N. However, a disadvantage possessed by employing these alloys used in the molten state is the high cost of the nitriding processes, i.e. providing nitrogen in the metals or alloys.

The nitrogen-enrichment (nitriding) of alloys to produce alloy nitriding agents, are produced in the solid phase in special furnace plant and are rather expensive besides involving high plant-investment costs they also consume considerable heat, usually in the form of electrically generated heat, together with large volumes of nitrogen.

It is the object of the invention to provide a method for producing effective nitriding agents which involves no significant investment cost due to the necessity of erecting special plant, and requires no additional supply of, for instance, electrical heat, besides consuming no major volumes of specially prepared nitrogen gas. According to the invention this is achieved by utilising, for nitriding crushed steel-nitriding agents, the heat from the exothermic process of producing cyanamide from calcium carbide, and by making use of the large surplus of nitrogen gas required in this exothermic cyanamide process.

It is well known that in the production of calcium cyanamide by the conventional methods, such as the Polzeniusz, or Frank and Caro processes, a considerable amount of radiant heat is generated by the exothermic reaction, and that a considerable amount of nitrogen gas is required, in excess of stoichiometrical quantities. This radiant heat and the excess nitrogen can be used according to the process of the invention for nitriding the required steel-nitriding agents.

According to a preferred feature of the invention for treating steel-nitriding agents in conjunction with the

cyanamide process, flat sheet metal trays or containers are, by means of suitable holding devices, suspended in or placed on the drums into which the calcium carbide is charged for the production of calcium cyanamide, and the agents that are to be treated are loosely charged onto the said trays or containers.

FIGURES 1 and 2 of the accompanying drawings illustrate a vertical and horizontal section of such an arrangement.

The calcium cyanamide is produced in drums **1** with a refractory lining and these are conveyed on trucks, of which the top **2** of one is shown in the drawing, through the cyanamide furnace. When the calcium carbide charge **3** has been charged into the drum **1**, shallow pans or trays **5** are placed onto the bracket-like supports **4** and filled with a thin layer **6** of the agent which is to be nitrided.

Alternatively, the crushed agent which is to be treated may be filled into containers of round or rectangular section. These are inserted into the calcium carbide from which the cyanamide is to be produced. The sides of the containers, which have an open top are perforated with a number of holes preferably of from 2 to 8 mm. diameter, to facilitate the entry of the nitrogen gas. Before the agent is introduced into the containers, the containers are lined with paper to prevent the crushed agent from falling out and from being contaminated with carbide. The outside of the containers may likewise be wrapped in paper to facilitate their subsequent extraction from the calcium cyanamide block produced in the main process.

FIGURES 3 and 4 of the accompanying drawings are a vertical and horizontal section respectively of such an arrangement.

So far as parts **1**, **2** and **3** are concerned the arrangement is similar to that shown in FIGURES 1 and 2. The agent which is to be treated is filled into the open top perforated cylindrical containers **7** or rectangular containers **8**, after these have been internally lined and externally covered with paper for the above mentioned reasons.

The treatment of the metal or alloy proceeds when the carbide has ignited and the exothermic nitrogen-fixing reaction has started, in that the agents in the sheet metal pans or containers embedded into the carbide is exposed to the heat of radiation generated by the reaction of the carbide to calcium cyanamide. The temperatures thus reached rise to a level at which the agents absorb nitrogen.

As its nitrogen content increases, the temperature of the metal or alloy also rises and the particles sinter together. At the end of the reaction a hard slab is formed in the sheet metal trays, or hard blocks are formed in the round or rectangular section containers. During this treatment the contents of the trays **5** and of the containers **7**, **8** must not make direct contact with the calcium carbide from which the cyanamide forms, in order to prevent an undesirable carburisation of the nitriding agents taking place.

In numerous runs performed in a cyanamide plant on an industrial scale, nitrogen contents of 4 to 18% were obtained in the agents thus treated, for instance a highly refined ferrochrome contains 5 to 7% of nitrogen, a highly refined ferromanganese contains 4.7 to 6.6% of nitrogen, a ferromanganese contains 3.5 to 4.5% of nitrogen, a chromium metal contains 4 to 6% of nitrogen, a ferroniobium-tantalum contains 4 to 6% of nitrogen, and in a manganese-vanadium containing 56 to 65% V and 24 to 26% Mn, the nitrogen content was as high as 14 to 18%.

Adherence to the following conditions has been found to be very useful for promoting the treatment according to the invention:

(1) The nitriding agents should be ground to a particle size not exceeding 1 mm., preferably below 0.25 mm.

(2) The depth of the layer **6** of comminuted agent on the sheet metal trays **5** should not exceed 100 mm., and preferably it should be not more than 60 mm.

(3) When the crushed agents are introduced in containers, round section containers may have a diameter of about 250 mm. and rectangular section containers may be between 50 and 100 mm. x 300 to 600 mm. The length of the containers which have an open top and a solid bottom may be ½ to ⅔ of the depth of the carbide charge **3**. With advantage the containers may be of divided construction to permit them to be conveniently opened when they have been extracted from the cyanamide block, and the treated agents to be easily removed. The side walls of these containers are perforated to facilitate the entry of the nitrogen gas. The insides of the containers are lined with paper and they are provided with a paper wrapping on the outside to prevent the agents from trickling out and the carbide or cyanamide from contaminating the agents.

(4) The quantity of the agent that is to be treated should constitute only a fraction of the carbide which is to be converted to cyanamide to ensure that the radiant heat developed during the cyanamide reaction is sufficient for nitriding the agents.

(5) At the end of the process of nitrogen fixation by the carbide the agents should be carefully taken out of the trays or containers and kept separate from the cyanamide that has been formed to prevent contamination of the nitrided sintered agents.

The method of nitriding metals or alloys to produce nitriding and alloying agents according to the invention can be performed in cyanamide plant in which either the Polzeniusz or the Frank and Caro process is performed, the heat radiated during the exothermic calcium cyanamide process and the nitrogen gas excess required for its performance being utilised. The investment cost is therefore limited to the provision of suitable sheet metal trays **5** and brackets **4** or of suitable containers **7** or **8**. The nitriding cost is very low since it makes use exclusively of surplus heat and surplus nitrogen gas from the cyanamide process, and consumes only paper for lining and wrapping the containers. The labour involved and the wear of trays or containers is low since suitable high

temperature steels for such a purpose are readily available.

The nitrided agents which have sintered together in the form of plates in the trays or in the form of blocks in the containers are comminuted to a size that can be conveniently handled by the metallurgist. The abraded fines are briquetted or added to fresh crushed agents and subjected together with them to another treatment according to the invention.

What I claim is:

1. A method of producing nitrogen-containing metal for use as a nitriding and alloying agent which comprises:

(1) comminuting a metal,

(2) confining said comminuted metal in a vessel,

(3) contacting said confined comminuted metal with the residual gases and the exothermic heat of reaction produced in a zone where calcium carbide is reacted with nitrogen present in stoichiometric excess amounts to provide calcium cyanamide, thereby producing a sintered nitrogen-containing metal, and

(4) crushing said sintered nitrogen-containing metal.

2. The method of claim **1** wherein said metal is a metal alloy.

3. The method of claim **1** wherein said metal is confined in a perforated vessel.

4. The method of claim **1** wherein the metal is comminuted to a grain size below 1 mm.

5. The method of claim **4** wherein the metal is comminuted to a grain size below 0.25 mm.

6. The method of claim **1** which includes (5) adding said crushed sintered nitrogen containing metal to additional comminuted metal and repeating steps (2)–(5) as long as desired.

7. The method of claim **3** wherein said perforated vessel is inserted in said zone free from direct contact with said calcium carbide.

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