July 2, 1963 E. E. V. HELIN 3,096,174

METHODS OF REDUCING A METAL OXIDE BY A CARBONACEOUS MATERIAL AT SUB-ATMOSPHERIC PRESSURES

Filed April 10, 1958

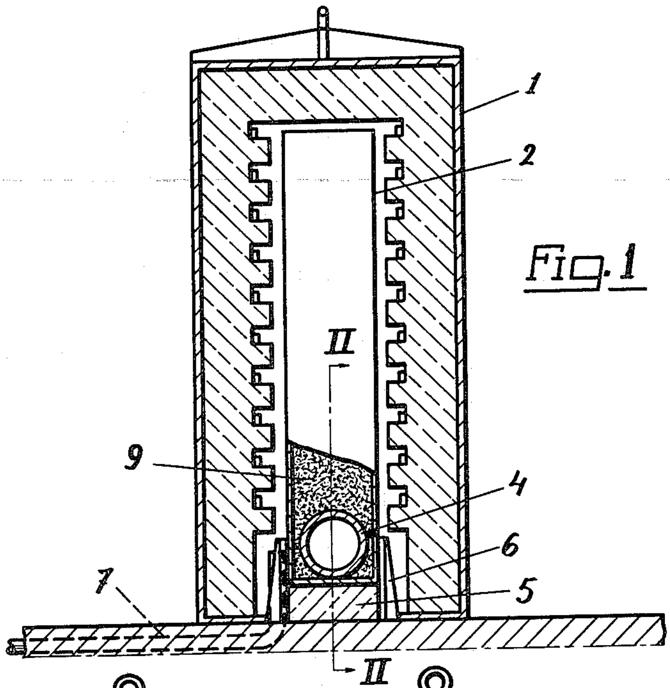

Fig. 1

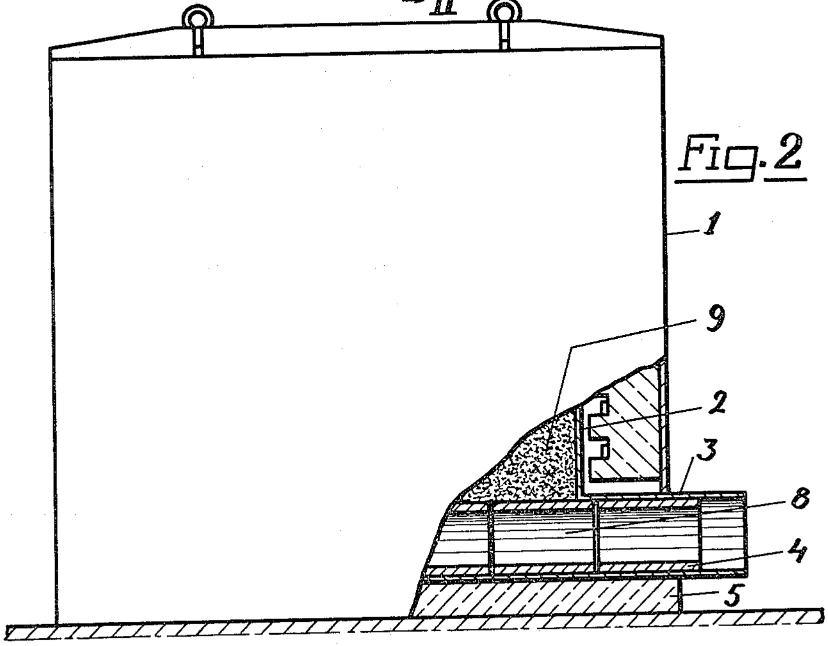

Fig. 2

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United States Patent Office

3,096,174
Patented July 2, 1963

3,096,174

METHODS OF REDUCING A METAL OXIDE BY A CARBONACEOUS MATERIAL AT SUB-ATMOSPHERIC PRESSURES

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Filed Apr. 10, 1958, Ser. No. 727,660
Claims priority, application Sweden Apr. 18, 1957
5 Claims. (Cl. 75—226)

This invention deals with processes involving the heat-treatment of finely comminuted materials under sub-atmospheric pressures, as well as with means for carrying out such heat treatment.

Examples of such processes are metal producing processes in which a mixture of metal oxide and carbon is heated at sub-atmospheric pressures to a temperature below the melting points of the metal oxide and of the metal produced. Another example is the decarburizing of powdered metals, particularly high-carbon ferrochromium, by the process of heating a mixture of the metal to be decarburized and an oxidizing agent, which latter may consist of a layer of oxide on the individual metal particles formed in a preceding oxidizing treatment. While the invention is primarily concerned with processes of the types just indicated, it is not limited thereto but may also be applied to processes in which a comminuted charge is to be heated at sub-atmospheric pressures in order to effect, for instance, a de-gassing of the charge or a distillation or a sublimation of desirable products therefrom or a removal of undesirable constituents.

At present, processes involving the heating in vacuo of a powdered material have found very limited practical application because of the high costs. Conventional vacuum furnaces for large charges are expensive to build and have a comparatively low production capacity, a long time being required both for heating the charge to the temperature required and for allowing the charge to cool to the temperature at which the furnace can be opened and the charge removed therefrom.

The invention has for its principal object to provide a process for the vacuum heating of powders which can be carried out at low cost and in which the processing time per charge is comparatively short. Another object is the provision of a process for the vacuum heating of powders in which the furnace proper is allowed to operate at atmospheric pressure. Another object is to improve the transmission of heat to the powder charge. Still another object is the provision of improved means for carrying out the process.

According to a principal feature of the invention, the finely comminuted material to be heat-treated is charged into a sheet metal container the wall thickness of which is insufficient to enable the container to retain its shape against the atmospheric pressure on exhaustion at elevated temperatures, whereupon said container with the charge contained therein are heated to the temperature required while a sub-atmospheric pressure is maintained in the interior of said container. It will be noted that, in this method, the furnace proper does not have to be operated at subatmospheric pressures. It will therefore be possible to employ a furnace of any of the types usually employed for metallurgical heat treating, for instance a resistance furnace or a gas-fired furnace. As soon as the powder charge has been submitted to the treatment temperature the required time, the container can be removed from the furnace and replaced by another container enclosing a new charge to be heated. In this manner, the furnace can be maintained continuously at its full operating temperature. The outer pressure will force the heated container wall inwards, so that a close engagement between the container wall and the powder charge will be main-

tained, resulting in an efficient transmission of heat from the furnace chamber to the powder charge throughout the heating period.

In this context, the expression "powder" or "comminuted materials" should be understood to comprise not only fine-grain powders, but also materials composed of comparatively large grains or particles, generally with a particle size up to ½ inch. If the powder to be heat-treated is fine-grained or contains a substantial proportion of fine grains, for instance with a particle size below 0.5 mm. (0.02 in.), and is of such a composition as to develop substantial quantities of gas on heating, it may be necessary or advisable to shape the powder, with or without the admixture of a binder, into pellets or other formed bodies, preferably of a size not exceeding ½ inch. According to another possibility, the powder with or without the addition of water or a binder is compressed or compacted in the container so as to form a more or less coherent, porous body which may be provided with one or more exhaust canals serving to facilitate the discharge of gases or vapours from the material during the subsequent heat treatment, which in this case may be preceded by a special drying treatment at lower temperatures serving to remove the water or solvent, if such have been employed.

Regarding the choice of the shape and size of the sheet metal container, it is to be noted that the distance from any individual powder particle to the nearest portion of the container wall should be short, if a swift heating of the entire charge to the temperature required is to be obtained. Preferably, therefore, the container is given the shape of a flattened case, the thickness of which may for instance be 5 in. or less.

In the accompanying drawings illustrating an embodiment of the invention,

FIG. 1 is a cross-sectional view of a furnace enclosing a sheet-metal container, and

FIG. 2 is a side view of the furnace, partially in section on the line II—II of FIG. 1.

The furnace **1** is an electric resistance furnace of the bell type adapted to be lifted and moved as required by means of a traversing hoist not shown. The furnace is shown in operative position enclosing a container **2** shaped as a flat case of thin sheet metal, for instance 1.5 mm. (0.059 in.) carbon steel sheet or silicon-chromium alloyed heat resistant steel sheet. The case is supported in an upright position on a stand **5** with side stanchions **6**. The case is provided with a vent **8** connected to an outlet tube **3** adapted to be connected to a vacuum pump (not shown). In the outlet tube and along the bottom of the casse are placed a number of ceramic sleeves **4**, the purpose of which is to form an exhaust canal and to protect the tube **3** against collapsing under the action of the outer pressure.

The powder **9** to be heat-treated, for instance a partially oxidized ferrochromium powder, is charged into the container from above and carefully packed by agitation of the container or by ramming. The lid is then welded on, the container is put in position on the stand, and the tube **3** connected to the vacuum pump. After the bell furnace has been lowered over the container, a protective gas is supplied to the furnace chamber through the tube **7**. As soon as the material under treatment has reached the required temperature or been held at high temperature for a required period, as the case may be, the bell furnace is raised and preferably put into operative position with regard to a charged container supported by another stand (not shown). To protect the first container against the oxidizing action of the air during the cooling period, a bell-shaped cover may be applied over the container as soon as the bell furnace has been removed. The supply of protective gas through the pipe **7** may be allowed to continue for part of or all of the cooling period.

Instead of the ceramic sleeves 4, it is possible to employ other means, for instance a row or heap of irregular pieces of ceramic material or of the same material as the powder charge, in which case a piece of metal netting or a perforated sheet may be applied on the row or heap to prevent the powder from filling the space between the irregular pieces.

It is also possible to employ a stationary furnace in the carrying out of the method according to the invention. The furnace may be adapted to the simultaneous heat treatment of two or more containers. The exhaust outlet may, if desired, be provided at the upper end of the container. To permit the removal of a container in red-hot condition without danger of oxidization of the powder charge, provision may be made for hermetically closing the container while still under sub-atmospheric pressure, for instance by flattening the outlet pipe and applying a seam weld across the flattened portion. Provisions may also be made for filling the container with a protective gas before removal from the furnace.

In some cases it may be useful to subject the comminuted material to a heat treatment in a gas of atmospheric pressure before or after the heat-treatment at sub-atmospheric pressure. For instance, the decarburizing heat treatment of ferro-chromium powder may comprise as a final step the filling of the container with hydrogen at atmospheric pressure in order to remove possible rests of carbon and/or oxide. If required, this step may be repeated twice or more, the reaction products being removed between the successive filling steps.

Before the treatment according to the invention, the charged container may be subjected to a rinse with protective gas, so that the air remaining in the container will be replaced by the protective gas.

In a particular method according to the invention, the container is exhausted and hermetically closed before starting the heat treatment. Conveniently, said steps are carried out on the container while still outside of the furnace. This particular method will, of course, be possible only when the charge has such a composition that the development of gas therefrom during the heat treatment will be insignificant. An example is the heating of containers of stainless austenitic chromium-nickel steel sheet filled with a powder of the same kind of steel produced by granulating or atomizing liquid metal with subsequent removal of the oxide layer on the grains or particles, for instance by mechanical treatment in a hammer mill. The stainless steel employed may advantageously contain a small proportion of boron, for instance 0.1% or less, in order to improve the ability of the grains to weld together into a coherent body.

I claim:

1. In the method of reducing a metal oxide by a carbonaceous material by the heating of a comminuted solid mixture of said materials at sub-atmospheric pressures, the steps of charging said unreacted mixture into a sheet metal container the wall thickness of which is insufficient to enable the container to retain its shape against external atmospheric pressure on exhaustion at elevated temperatures, and heating said charged container at atmospheric pressure while maintaining a sub-atmospheric pressure in the interior of said container.

2. In the method of reducing a metal oxide by a carbonaceous material by the heating of a comminuted solid mixture of said materials at sub-atmospheric pressures, the steps of charging said unreacted mixture into a sheet metal container the wall thickness of which is insufficient to enable the container to retain its shape against external atmospheric pressure on exhaustion at elevated temperatures, exhausting said container so as to produce a sub-atmospheric pressure therein, hermetically closing said container while maintaining said sub-atmospheric pressure therein, and heating said hermetically closed container in a furnace chamber maintained at atmospheric pressure.

3. In the method claimed in claim 1, the further steps of compressing the mixture in the container prior to the exhaustion thereof, and of providing at least one exhaust canal in the body of compressed or compacted material.

4. In the method as claimed in claim 1 the further step of shaping the comminuted mixture into pellets before charging the mixture into the container.

5. In the method as claimed in claim 1, the further step of compacting the mixture in the container prior to exhaustion thereof.

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