APPARATUS FOR THE MANUFACTURE OF CHROME POWDER

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9 Claims

Abstract of the Disclosure

An apparatus for the manufacture on an industrial scale of chromium powder from chromium oxide powder by treating the latter with magnesium vapor. The apparatus comprises a bell-type furnace heated by external electrical resistors, one or more columns of crucibles, each adapted to receive a reaction mass of substantially 1 to 10 kg. of chromium oxide powder into which a magnesium ingot has been embedded. A heat-insulating material is interposed between two adjacent superimposed crucibles.

The invention relates to a process and an apparatus for the manufacture of chromium powder.

It has already been proposed to manufacture chromium powder by subjecting chromium oxide powder to the action of magnesium in vacuum form. In this way, starting from fine chromium oxide powder, sometimes called chromium green, chromium powder of a previously unobtainable degree of fineness and of great purity is obtained.

The object of this invention is to extend the process on an industrial scale, i.e., to make it possible to treat in a single operation masses of chromium oxide of sufficient bulkiness for the cost to permit numerous and wide applications.

A particular object of the invention is to provide a process and an apparatus for obtaining in a single operation chromium powder in several dozen kilograms or even more in spite of the highly exothermic character of the reaction.

Another object of the invention is to provide a process and an apparatus whereby an exothermic reaction can be controlled satisfactorily without prolonging excessively the reaction time. A general object of the invention is to provide a process and an apparatus for obtaining chromium powder of a much greater degree of fineness than that of ordinary industrial powder and at a competitive cost.

The process involves using magnesium in the most available form, i.e., in the form of ingots, and disposing the powder to be treated in the same receptacle as the ingot or ingots, so that the working of the process is particularly simple.

The invention comprises steps that enable violence of the reaction to be effectively counteracted. This violence is liable to occur because of the exothermic character of the reaction. By these steps the temperature of the whole is limited to the desired value so that the vaporization of the magnesium is not accelerated beyond what is necessary.

According to the invention, for the industrial treatment of chromium oxide powder with magnesium in vapour form in order to obtain chromium powder, an industrial quantity of chromium oxide powder is divided into masses between one and about 10 kilograms, magnesium in iron-got form is embedded in each of these masses, which are thermally insulated from each other, and the industrial quantity is heated in the same enclosure and in a single treatment phase.

Thus each portion of the mass is brought into the optimum reaction conditions without being subjected excessively to the influence of the heat liberated by the other portions simultaneously undergoing the reaction. According to the invention, then, the process resulting in the production of chromium oxide powder is influenced by dividing the treated mass.

The invention makes it possible to take advantage simultaneously of the bulk of the mass treated in one operation, with the resulting economic advantages, and also the strictness of the control to which each of the portions can be subjected so that the reaction may proceed under optimum conditions, with, as a result, a complete or practically complete conversion of the chromium oxide into chromium of exceptionally fine particle size.

In particular, the invention avoids the formation, in spite of the bulk of the total mass treated, of conglomerates of various compositions resulting from excessive local raising of the temperature. These conglomerates otherwise appear inevitably when chromium oxide in a large mass is heat-treated with magnesium vapour.

The invention therefore provides a means for extending the industrial use of magnesium as a reducing agent for oxides, such as \( \text{Cr}_2\text{O}_3 \), whose formation heat is not so great as that of the oxides usually reduced by magnesium, while keeping the reduced metal in the state of a very fine powder.

In one embodiment of the invention, the mass of chromium oxide to be treated is distributed in crucibles or pots piled one on top of each other, means being provided for the provision of thermal insulation between the successive crucibles.

To provide this thermal insulation, the invention proposes the application of an insulating layer as a heat barrier between the portions contained in two successive crucibles. For this purpose it proposes, for example, divided magnesia or alumina in the form of a powder of sufficiently porous agglomerate.

The invention is also characterised by the use of graphite as material to make the crucibles intended to be superimposed on each other, which retain their form in spite of the mechanical stresses and thermal influences to which they are subjected, so that they can be disposed in a column for the treatment of large masses.

The invention also relates to an arrangement whereby the graphite crucibles are disposed in several columns; the high conductivity of the graphite ensures a good distribution in the reaction masses of the thermal flux coming from one or several peripheral resistors.

The invention also proposes placing the reaction mass in iron or mild-steel dishes because of their resistance to corrosion by magnesium, each of the dishes being placed in a graphite crucible.

The crucibles are in an inert atmosphere, for example of argon, leak-proofness being ensured by a cooled seal remote from the treatment crucibles.

In the following description given by way of example reference is made to the accompanying drawings in which:

- FIG. 1 is a diagrammatic vertical section through an apparatus according to the invention in one embodiment;
- FIG. 2 is a diagrammatic section through a crucible according to the invention in one embodiment;
- FIG. 3 is a view similar to FIG. 2 but for another embodiment; and
FIG. 4 is a diagrammatic horizontal section through another embodiment of the apparatus according to the invention.

With reference to FIG. 1, the furnace 10, which is raisable and lowerable, has a wall 11 made of refractory material on whose inner face there is a heating resistor 12, which is advantageously divided into sections; the current supply to each section may be controlled independently by means indicated diagrammatically at C. In FIG. 1, the furnace rests on the bottom 13, thus bounding a cylindrical space 14. The treatment enclosure 15 is bounded by a bell-shaped member 16, made for example of refractory alloy and of a generally cylindrical shape, being so positioned at the bottom edge of the bell-shaped member being provided by a known array of ribs, rubber or the like and cooled by water circulation.

Crucibles or boxes 18a, 18b, 18c, etc., for example about ten in number, are piled one on top of the other, the crucible 18 resting by its bottom 19 on the top edge 20 of the crucible 18a, etc. The bottom crucible 18 rests on a base 21, keeping it away from the bottom of the furnace and the seal 17. Each of the crucibles 18 is made of graphite and contains in its interior an iron or mild-steel dish 22.

Heat insulation is provided between two adjacent crucibles. In the embodiment described heat insulation 24 is provided between the bottom 19 of each crucible, with the exception of the bottom crucible 18, and the bottom 23 of the dish it contains. This insulation may consist of a layer of powdered alumina or magnesia. As a modification, a disc may be provided that is formed by an agglomerate of a material that is sufficiently insensitive to magnesia vapours, such as magnesia or alumina, and has sufficient porosity to ensure good heat insulation. A porous-carbon disc may also be used.

A magnesium ingot 25 embedded in chromium oxide powder to be treated 26 is placed in each of the receptacles 22. The chromium oxide powder is commercial powder. Its fineness depends on the fineness of the chromium powder it is desired to obtain. The quantity of chromium oxide powder contained in each crucible may be between 1 and about 10 kilograms, and the magnesium mass accounts with the quantity of chromium oxide powder.

Argon, which forms the protective atmosphere, arrives at the bottom of the furnace through the passage 27. The current supply of each section of the heating resistor 12 is controlled independently from a thermostatic element that is sensitive to the temperature of the reaction mass of the crucible or crucibles opposite the resistor section and therefore mainly heated by the latter. This being a provision may be, for the different crucibles, for the observation of a profile in stages, for example in stages, of the variation of the temperature according to the time to which corresponds the progress desired for the reaction, which starts at about 800° C.

A programmer provides in known manner for the control of the electricity supply of the various resistor sections taking account of the control provided by the thermostatic elements.

During heating, the graphite crucibles maintain their shape without creep in spite of the high temperatures they endure; they may be used for a considerable number of treatments.

When submicronic chromium oxide powder is used as starting material, chromium powder is obtained whose particles have an average diameter approximately between a fraction of a micron and several microns.

In the embodiment shown in FIG. 2, an iron or mild-steel dish 40, the bottom of which 41 is convex, for example spherical, contains the reaction mass and rests on a layer 24 of powdered refractory material.

In the embodiment of FIG. 3, the dish 22, which has a substantially plane bottom, rests on a slab 42 made of sintered magnesia and held by an inner rim 43 of a graphite dish 44, the bottom of which thus has a large circular aperture 45.

In a variant of the apparatus according to the invention, the pots or crucibles may be placed side by side but separated thermally from each other. In this case heating is advantageously effected by the roof, the bottom or the roof and the bottom at the same time.

FIG. 4 relates to an apparatus according to the invention having four columns of crucibles 31–34, each of which is similar to the one described above with reference to FIG. 1. A central post 32 is provided; it may have partitions distributed regularly in the form of a cross with inwardly curved arms. The post and partitions are made of one or several materials adapted to effect good heat insulation. The crucible columns are thus thermally separated from each other.

The invention provides for the treatment with magnesia vapours of chromium oxide powders whose various particles are further away from each other than those of an ordinary chromium oxide powder. This promotes the action of the magnesia vapour and makes it more regular.

To obtain such a "blown" or "thinned" powder, it is proposed to subject ordinary chromium oxide powder to turbining in a turbine without a screen. This operation reduces the apparent density from 20% to 50%. The resulting emission of chromium oxide powder in air has enough stability for it to be used with advantage several hours after its formation.

What is claimed is:

1. An apparatus for the industrial manufacture of chromium powder by reduction of chromium oxide powder by magnesium in vapor form comprising: a bell-type enclosure, means for heating said enclosure disposed externally thereof, a plurality of superimposed crucibles each adapted to contain a reaction mass of substantially 1 to 10 kilograms of chromium powder, and an embedded magnesium ingot, and a layer of heat-insulating material interposed between the reaction masses of two adjacent superimposed crucibles.

2. An apparatus as claimed in claim 1 wherein the layer is made of a powder of a refractory heat-insulating material selected from the group comprising alumina and magnesia, said powder layer being interposed between the bottom of the crucible and the receptacle containing the reaction mass placed in the latter.

3. An apparatus as claimed in claim 2, wherein said crucibles are made of graphite and said receptacles are made of a metal selected from the group comprising iron and mild steel.

4. An apparatus as claimed in claim 1, wherein the bottom of each crucible has a large central aperture, and a disc of heat-insulating material resting upon said aperture.

5. An apparatus as claimed in claim 1, wherein said heating means comprises electrical resistors divided into a plurality of groups located one above the other and forming separate heating zones, the apparatus further comprising heat-sensing elements opposite said crucibles, and means connected to said resistors and to said elements for controlling the temperature of said resistors as a function of signals delivered by said sensing elements during the reduction reaction.

6. An apparatus as claimed in claim 1, wherein said crucibles are piled one on top of another to form a multiplicity of columns, and heat-insulating means between said crucible columns to insulate the same from one another.

7. An apparatus in accordance with claim 6, including a centrally disposed post, said columns being disposed around said post.

8. Apparatus in accordance with claim 7, wherein said post is shaped for partially receiving said crucible columns, and is formed of material which thermally insulates the columns with respect to each other.
9. Apparatus in accordance with claim 4, said disc being formed of a porous agglomerate selected from the group comprising sintered magnesia and porous carbon.

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