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PROCESS FOR PRODUCING PURE MANGANESE

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The present invention which is based upon the results of applicants' researches, has for its object the production of pure manganese from alloys of manganese with other metals by vaporization in a vacuum.

Ferromanganese alloys having a more or less high manganese content, and generally containing carbon, are produced industrially in an electric furnace or a blast-furnace.

Experiments have shown that manganese when alloyed with iron and carbon can easily be vaporized at a definite temperature at a pressure which can be deducted from a curve which passes through the following points:

0.01 mm. Hg at 1000° C.
0.06 mm. Hg at 1100° C.
0.3 mm. Hg at 1200° C.
1.2 mm. Hg at 1300° C.

This curve approximates closely the vapor pressure curve of pure manganese.

This means, on the one hand, that the energy binding the manganese and the other elements in the Mn/Fe/C alloys is slight and, on the other, that manganese exhibits great reactivity in these alloys.

In order to increase the vapor pressure of manganese above the alloys, it has been instinctively attempted to increase rapidly the temperature and, in particular, to work above the melting point. It has been established experimentally that, under these conditions, the practical vaporization speed becomes exceedingly small.

The process which is the object of the present invention consists in producing pure manganese by vaporizing, without any apparent melting, the alloy serving as the raw material which is supplied in the form of granules, for example, 5 to 20 millimeters in size, the granules being preserved as individual units until the manganese has been completely extracted.

To attain this result, the vaporization should be initiated at a temperature lower than the melting point of the raw material, so that each granule becomes coated with a layer of the porous graphitic residue left behind by the evolved (departing) manganese. The temperature is then progressively raised up to the melting point and beyond, the molten material remaining enclosed inside each granule by the graphitic residue. The vaporization surface thus remains practically unchanged throughout the entire process, the porous graphitic residue acting as a wick for the product which it encloses.

The melting point of the alloy varies from about 1100° C. for some ferromanganese alloys, to about 1250° C. for the richest manganese carbides.

A characteristic feature of the invention consists in adopting a stagewise operation at successive temperature levels, for example,

6 hours at 1100° C.
6 hours at 1150° C.
6 hours at 1200° C.
6 hours at 1250° C.

the pressure being necessarily less than 0.06 mm. Hg during the 1100° C. heating stage in order to enable the desired volatilization to take place, and being subsequently increased with the temperature, while remaining lower than the values on the pressure curve mentioned above. Obviously, the example of temperature increases given above is not limiting in any way; the melting point of the particular charge is the only factor which determines the first temperature level which, of course, must be lower than said melting point. In this way, the practical extraction speed, which is the higher the lower the working pressure, will remain throughout the process at a value compatible with the requirements of a commercial plant, this being aided by the increase in temperature.

Example 1

100 kg. of carbon-containing manganese in the form of granules 5 to 20 mm. in size and containing

	Percent
Mn	88
Fe	4
C	7.5
Si	0.5

are shovelled upon the hearth (0.75 m.²) of a 40 kw. electric vacuum furnace. After the furnace has been closed and placed, by known means, under a vacuum of 0.02 mm., there is carried out the stepwise increase of temperature described above.

The manganese which is condensed in compact form on a cooled iron tube, can be taken out of the furnace while the latter is still hot (1000° C.). The condensed manganese weighs 83.1 kg. and contains

	Percent
Mn	99.9
Fe	0.005
Si	0.01
C	0.04
Cu	0.04

other metals being present as traces.

The residue which weighs 11.8 kg. and each granule of which has retained the shape and volume of the initial alloy granules, is easily removed by means of a scraper.

The extraction rate of the manganese is nearly 100% and the condensation yield is approximately 95%.

Example 2

100 kg. ferromanganese granules 5 to 20 mm. in size and containing

	Percent
Mn	78
Fe	14.5
Si	0.5
C	7

are charged on the hearth of the same furnace as in Example 1. The increase in temperature is carried out under the conditions of Example 1, and there are recovered 65 kg. of a condensate containing

	Percent
Mn	99.85
Fe	0.02
Si	0.01
Al	0.02
Pb	0.02
Cu	0.02
P	0.01
S	0.01
C	0.04

other elements being present as traces.

The residue weighs about 30 kg. and still contains a

small quantity of alloyed manganese. However. the extraction rate is nearly 90% and the condensation yield, i. e.

$$\frac{\text{condensed Mn}}{\text{charged Mn}}$$

is approximately 83%.

The metal produced in this way is an extremely pure refined manganese which can advantageously replace manganese prepared by electrolysis in its various applications.

We claim:

1. In the process of producing pure manganese by evaporation in a vacuum from a carbon-containing manganese alloy, the improvement in carrying out said process which consists in evaporating the manganese from the particles of said alloy in a plurality of stages at progressively increasing temperatures, the first stage being maintained at a temperature below the melting point of the alloy, and condensing and collecting the manganese so evaporated.

2. The process of producing pure manganese from a carbon-containing manganese alloy, which comprises the steps: heating particles of said alloy in a vacuum to a temperature below the melting point of said alloy but sufficient to evaporate manganese therefrom, whereby a porous graphite layer is formed on each individual particle; condensing and collecting the manganese so evaporated; thereafter raising the particles to a higher temperature, maintaining the particles at said higher temperature whereby additional quantities of manganese are evaporated, and condensing and collecting such additional evaporated manganese.

3. The process of producing pure manganese from a carbon-containing manganese alloy, which comprises the steps: heating particles of said alloy in a vacuum to a temperature below the melting point of said alloy, but

sufficient to evaporate manganese therefrom, whereby a porous graphite layer is formed on each individual particle; condensing and collecting the manganese so evaporated; thereafter heating said particles to, and maintaining them at, progressively higher temperatures including temperatures above the melting point of the alloy, whereby the particles remain in their individual state without coalescing, and molten alloy migrates by capillarity through the porous graphite layer to the surface thereof where further quantities of manganese are evaporated from the alloy; and condensing and recovering the additional manganese so evaporated.

4. A multi-stage process of producing pure manganese which consists in subjecting discrete particles of a carbon-containing manganese alloy to heating in a vacuum according to a heating schedule which comprises (*a*) an initial heating to a temperature below the melting point of the alloy, and (*b*) at least one subsequent heating to a temperature above the melting point of said alloy; successively maintaining the alloy at said several temperatures whereby manganese is evaporated from the alloy; condensing and recovering the manganese so evaporated.

5. Process according to claim 1 wherein the initial heating is at temperature of about 1100° C. and a pressure less than about 0.06 mm. Hg.

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