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PROCESS OF MAKING A FERROALLOYING MATERIAL AND PRODUCT OBTAINED THEREBY

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This invention relates to a process for making a ferroalloying material and to the product produced by the process. More particularly, the invention relates to a vacuum furnace process for making a low carbon ferroalloying material containing at least two alloying metals one of which is derived from (a ferroalloy of) the group of ferroalloys consisting of ferrochromium, ferromanganese, ferrotitanium, ferromolybdenum, ferrotungsten, ferrovanadium, ferrozirconium and ferrohafnium and the other of which is obtained from a material containing an oxide of nickel or cobalt. Mixtures of the ferroalloys and of the metal oxides may, of course, be employed in the process.

The invention also relates to a ferroalloying material that is especially useful in making alloy steels and other ferrous alloys.

An object of the invention is to provide a process for the production of new ferroalloying materials including in addition to iron at least one of the metals nickel and cobalt and at least one of the metals chromium, manganese, titanium, molybdenum, tungsten, vanadium, zirconium and hafnium.

A further object of the invention is to provide a process for the production of the ferroalloying materials described in the immediately preceding paragraph that involves a vacuum furnace treatment that lends itself to simple, efficient and economical practice.

Another object of the invention is to provide a ferroalloying material that is low in carbon and hence is highly desirable for use in the manufacture of high alloy steels.

Another object of the invention is to provide an alloying product that is readily soluble in steel baths whereby the alloying materials are easily incorporated in steel melts in open hearth furnaces.

Another object is to provide a composite ferroalloying material whereby two or more alloying metals may be added to a steel bath in a single operation.

Another object is to provide a product that may be reduced to powder. The powdered material may be compounded with other materials to form an exothermic product that may be added to steel in the ladle. Exothermicity may be supplied, for example, by a silicothermic reaction in which the reactants may be silicon and sodium nitrate.

The foregoing and other aims, objects and advantages of the invention as may appear or be inferred from the following text are realized in a process for making a ferroalloying material which comprises reacting in contact with each other (1) a metal carbide such as medium to high carbon-containing ferroalloys of chromium, manganese, titanium, molybdenum, tungsten, vanadium, zirconium or hafnium or mixtures thereof or inter-alloys of the same with (2) a material containing substantial amounts of nickel oxide or cobalt oxide or mixtures thereof. The reaction is carried out by heating the materials in a confined zone under conditions of subatmospheric or low, absolute pressure to a temperature

and for a time sufficient to effect reaction between the oxide-containing material and the carbon-containing material with substantial reduction of the oxide-containing material to metal and substantial oxidation of the carbon of the carbon-containing material to carbon monoxide. Carbon monoxide or a mixture thereof with carbon dioxide and other gaseous impurities and products is withdrawn, preferably rapidly, from the confined zone. When the reaction is complete a metallic residue remains in the confined zone that is low in carbon and includes nickel and/or cobalt plus one of the metals chromium, manganese, titanium, molybdenum, tungsten, vanadium, zirconium and/or hafnium in addition to iron. The product also may contain small amounts of silicon and minor amounts of oxide impurities such as SiO_2, CaO, MgO, Al_2O_3 and the like.

In carrying out the process, the ferroalloy that contains carbon is comminuted as by grinding to fine particle size. The particle size of the material may be reduced to a point where substantially all of the material passes through a 100 mesh screen with a substantial portion of the grind being small enough to pass a 200 mesh screen. The metal oxide, such as nickel oxide, if not already of sufficiently fine size is reduced as by grinding so that most of the material passes through a 100 mesh screen.

Taking into account the carbon content of the ferroalloy and the nickel oxide and the purity of the nickel oxide-containing material, a mixture of the finely ground ferroalloy and nickel oxide-containing material is prepared wherein the amount of nickel oxide is so calculated with respect to the quantity of ferroalloy that sufficient oxide is provided to oxidize all of the carbon of the ferroalloy to carbon monoxide. A slight excess of nickel oxide may be incorporated in the mixture and sufficient excess may be incorporated to provide for oxidation of the silicon of the ferroalloy plus other minor ingredients capable of oxidation.

It is desirable to form the mixture into agglomerates or briquettes and for this purpose a binding agent is incorporated with the mixture. Any suitable organic or inorganic binder may be used. It has been found that a binder known as "Goulac" is suitable. "Goulac" is a powdered binder derived from waste sulphite liquor and includes lignin sulphonate as a principal binding ingredient. The amount of binder that is used may be about 2% by weight of the mixture, but amounts of binder ranging from ½% to 4% may be used. It is desirable to use a minimum quantity of binder and the amount of any particular binder that is employed is readily determined by simple experimentation.

The mixture is formed into agglomerates as by subjecting the mixture to a roll-pressing operation to provide agglomerates of proper size. The size of the agglomerates may range from one-half inch to two or three inches more or less. After pressing, the agglomerates are dried, if necessary, at a low to medium drying temperature.

Where Goulac is used as binder, the dried agglomerates may be heated to a somewhat higher temperature of say 450° C. to partially decompose and drive off part of the binder from the agglomerates. This operation is performed without vacuum so that the gaseous products are not carried through the vacuum system.

The decarburizing reaction is carried out in a confined zone such as is provided in the interior space of a vacuum retort. Suitable vacuum pumping equipment is provided to maintain the necessary low pressures in the retort.

Depending upon the size of the vacuum retort, a charge of agglomerated reaction mixture of suitable size is introduced into the retort and the retort is thereafter closed and placed under vacuum. If not already hot,

the retort is heated to the desired reaction temperature and kept at reaction temperature for the time necessary to decarburize the mixture under the vacuum conditions imposed. It is desirable that the vacuum system have ample capacity to withdraw the gases evolved in the reaction at a rapid enough rate to maintain the pressure in the reaction vessel at suitable low values. It is desirable that the pressure in the vacuum retort be reduced as rapidly as possible to about 40 to 50 mm. of mercury. It is further desirable that the final pressure in the vacuum retort when the last increments of carbon monoxide are being withdrawn be reduced to a value of the order of 150 microns of mercury or less.

Temperatures of the order of 1200° C. are suitable to promote the decarburization of the mixture. However, temperatures ranging from 1000° C. to 1400° C. may be employed.

The time of reaction will vary widely depending upon the agglomerates employed and the size of charge. Initial time to heat-up and out-gas the charge may range from one hour to a day or two. Operation at or below pressures of 20 to 50 mm. may continue for a period of a few hours to several days.

It is desirable to cool the residue after the reaction is complete and before exposing the residue to the atmosphere, this in order to eliminate or minimize oxidation of the residue. Preferably the retort and residue are cooled to a temperature below about 400° C. before the retort is opened and the residue exposed to the atmosphere. Below about 400° C. the residue may be removed from the retort and cooled in the atmosphere without undergoing excessive oxidation.

The residue from the vacuum furnace operation in general maintains its agglomerated form. Although some shrinkage does occur, the briquettes retain their original shape without breaking into finer pieces or particles which would not be desrable in the final product. The agglomerates are sintered, porous, dense aggregates of finely divided particles that are low in carbon and, in addition to iron from the ferror-alloy they contain the alloy metal or metals, such as chromium of the ferroalloy and a metal such as nickel and/or cobalt of the oxidant. The residue agglomerates are high in metallic content, low in slag content and are low to extremely low in carbon content. Carbon analyses less than 0.10% are readily attained and carbon analyses as low as 0.01 to 0.02% may be attained.

The residue agglomerates may be incorporated directly into steel for the manufacture of alloy steels in the open hearth or electric furnace.

Although the residue agglomerates are non-friable in ordinary handling and shipment, they are sufficiently friable that they may be converted to a powdered material by conventional methods. This powdered material may be made into an exothermic composition by addition of powdered ferrosilicon and sodium nitrate in proper proportions. Any other desired exothermic formulation may include the powdered alloying material of the invention. Such exothermic mixtures are eminently suitable for making ladle additions to steel.

Typically, the ferroalloying materials of the invention are in the form of sintered, porous, dense aggregates of finely divided particles containing a metal of the group consisting of nickel and cobalt in amounts rangng from 20% to 50%, a metal of the group consisting of chromium, manganese, titanium, molybdenum, tungsten, vanadium, zirconium and hafnium in amounts ranging from 25% to 65%, and carbon in amounts ranging from 0.01% to 0.15%, the alloying metal content of these aggregates being greater than 45%, the balance being substantially all iron except for incidental metallic and metallic oxide constituents.

In operating the vacuum furnace for the decarburization of the reaction mixture, it is important that, after initial out-gassing of the charge, the pressure should be, insofar as possible, continuously lowered throughout the course of the reaction to its final end value of 150 microns, more or less. By assuring continuous withdrawal of carbon monoxide from the aggregates in the vacuum retort, the pores of the aggregates are kept open and the final desired low carbon analysis is realized. By maintaining continuous flow of gas from the retort and continuously lowering the pressure, this desired result is achieved. On the other hand, if gas is not continuously withdrawn throughout the reaction period, the pores of the aggregates are likely to become sealed off and thereafter the reaction rate is reduced to a low order and the final carbon analysis is too high.

The principal reaction that takes place in the vacuum retort is summarized, for example, in the following equation:

$$(Fe_xCr_y)_zC + NiO \rightarrow NiCO + Z(Fe_xCr_y)$$

The mechanism of the reaction is not fully understood. Without wishing to be bound by any theory, it is believed that the nickel or cobalt oxide rapidly gives up its oxygen to the metal such as chromum of the high carbon ferroalloy with concomitant reduction of the nickel to the metallic state. Thereafter, the chromic oxide so formed reacts at a slower rate with the carbon of the ferroalloy to yield carbon monoxide that is evolved as a gas and withdrawn. In this stage of the reaction the chrome of the chromic oxide is reduced to the metallic state. The net reaction appears to be that given in the equation above.

The invention will be described with greater particularity in the following detailed examples.

Example I

The materals employed in this example are as follows:

(1) High Carbon Ferrochromium:

	Percent
Cr	53.04
Fe	32.24
Si	4.34
C	7.01

(2) Nickel Oxide:

	Percent
NiO	90+

(3) Goulac.

(4) Glucose.

The nickel oxide and the high carbon ferrochromium are separately ground to pass a 200 mesh screen and the materials are mixed in the following proportions:

	Pounds
HC FeCr	100
NiO	70
Goulac	3.5
Water	2.6
Glucose	2.0

The intimate mixture is briquetted in a Komarek-Greaves Rotary Press to provide pillow blocks measuring approximately 1" x 1" x 2". The green blocks are dried in a warm oven at 100° C. for a period of a few minutes to form a dried skin thereon that enables the blocks to be handled without breakage in subsequent furnace operations. The dried blocks are then preheated to 450° C. and held at this temperature for a period of from 1 to 2 hours to complete the drying of the blocks and to drive off the bulk of the Goulac. The briquetted blocks are charged to a vacuum retort tube which is subsequently raised to an operating temperature of 1200° C. Prior to heating, the retort is sealed and vacuum is applied.

Thereafter the charge is heated for a period of ten hours, the vacuum being maintained throughout the reaction period beginning at 45 mm. of mercury and continuously being reduced until at the end of the reaction

period of pressure is 80 microns and gas evolution is substantially nil.

The retort is allowed to cool for a period of 24 hours to a temperature of 350° C. The residue is then discharged into the atmosphere and allowed to cool to normal room temperatures.

The blocks although somewhat shrunken in size retain the general shape of the blocks as charged to the retort. They are metallic in appearance and exhibit a microscopic porosity. They are hard and dense.

147 lbs. of residue is obtained analyzing:

	Percent
Cr	34.98
Ni	34.95
Fe	22.84
Total Si as SiO_2	5.86
C	0.027

Example II

The materials employed in this example are the same as those used in Example I except that cobalt oxide, CoO, is substituted for nickel oxide. The materials are mixed in the following proportions:

	Pounds
HC FeCr	100
CoO	70
Goulac	3.5
Water	2.6
Glucose	2.0

The mixture is briquetted, dried and subjected to a vacuum furnace treatment in the same manner as the mixture of Example I. The vacuum furnace product is similar in appearance to that obtained in the immediately preceding example. 149 lbs. of vacuum furnace residue is obtained having the following analysis:

	Percent
Cr	34.90
Co	34.99
Fe	23.06
Total Si as SiO_2	5.57
C	0.049

Example III

This example illustrates large scale production of low carbon iron-chromium-nickel alloying material.

The materials employed are as follows:

		Percent
(1)	High carbon ferrochromium:	
	Cr	53.04
	Fe	32.24
	Si	4.34
	C	7.01
(2)	Nickel oxide:	
	NiO	99+
(3)	Goulac.	

The high carbon ferrochromium and the nickel oxide are ground to pass a 100 mesh screen with 70% through a 200 mesh screen.

The materials are intimately mixed in the following proportions:

	Pounds
HC FeCr	6,880
NiO	4,820
Goulac	241
Water	179

The mixture of ingredients is briquetted, dried and heated to about 450° C. to drive off most of the binder and all of the residual water.

The dried briquettes are charged into a vacuum retort internally heated by electric resistance elements, the retort being at room temperature. The retort is closed and vacuum is applied. The heating elements of the retort are energized and the charge comes up to a temperature of 1200° C. in 24 hours at which time the pressure within the retort is 2,000 microns. The furnace temperature is maintained at 1200° C. for an additional period of 106 hours; the furnace pressure is still 2,000 microns at the end of 26 hours. At the end of 46 hours the furnace pressure is 800 microns. The furnace pressure drops to 250 microns at the end of 75 hours and to 50 microns at the end of 94 hours. A pressure of 50 microns is maintained in the furnace until the power is cut off at 106 hours. Thereafter the furnace is allowed to cool for an additional period of 95 hours and is opened to the atmosphere at a temperature of 320° C. and discharged.

The residue is in the form of the original briquettes that have shrunken in volume to approximately one-half of the volume of the briquettes as charged. They have a bright metallic luster, are hard and dense and have a microscopic, porous appearance.

The vacuum furnace residue weighs 10,000 lbs. The residue has the following analysis:

	Percent
Cr	35.14
Ni	34.89
Fe	23.24
Total Si as SiO_2	5.03
C	0.043

Example IV

This example is typical of the production of a low carbon iron-manganese-nickel alloying material from high carbon ferromanganese and nickel oxide.

The materials are as follows:

		Percent
(1)	High carbon ferromanganese:	
	Mn	80.67
	Fe	11.47
	Si	0.235
	C	6.87
(2)	Nickel oxide:	
	NiO	99+
(3)	Goulac.	

After grinding the high carbon ferromanganese and nickel oxide to pass a 200 mesh screen, the materials are mixed as follows:

	Pounds
HC FeMn	100
NiO	44
Goulac	3
Water	2

The mixture of ingredients is briquetted and dried. The dried briquettes are placed in a vacuum retort for a period of 12 hours at a temperature of 1200° C. under an initial vacuum of 500 microns of mercury and final vacuum of 60 microns of mercury. At the end of this period, gas evolution has subsided to a very low value.

The retort is cooled and the contents discharged. The residue weighs 131 lbs. and has the following analysis:

	Percent
Mn	65.30
Ni	25.65
Fe	8.50
Si as SiO_2	0.46
C	0.083

The residue is metallic, porous and hard.

From the foregoing description it will be seen that the present invention provides a very useful process for producing low carbon ferroalloys that find ready application in the production of alloy steels.

I claim:

1. A process for making a three-metal furnace additive for use in making stainless steels which comprises reacting in the solid state a material containing a substantial proportion of a metal oxide selected from the group consisting of nickel oxide and cobalt oxide in intimate

contact with a ferroalloy material containing a substantial amount of carbon selected from ferroalloys of the group consisting of ferrochromium, ferromanganese, ferrotitanium, ferromolybdenum, ferrotungsten, ferrovanadium, ferrozirconium, ferrohafnium, and mixtures and composite alloys thereof by heating said materials in a confined zone under conditions of low absolute pressure to a temperature below the point of substantial fusion of said materials and for a time sufficient to effect reaction between the metal oxide-containing material and the ferroalloy with substantial reduction of the metal of the metal oxide to the metallic state and substantial oxidation of the carbon of the ferroalloy to an oxide of carbon, and withdrawing the oxide of carbon formed in the reaction from said confined zone, whereby to yield a metallic residue low in carbon including the metal of the metal oxide-containing material and the metals of the ferroalloy substantially all in the metallic state.

2. A process for making a three-metal furnace additive for use in making stainless steels containing iron, nickel and chromium which comprises reacting in the solid state nickel oxide with ferrochromium containing a substantial amount of carbon by heating an intimate mixture of said nickel oxide and said ferrochromium in a confined zone under conditions of low absolute pressure to a temperature below the point of substantial fusion of said materials and for a time sufficient to effect reaction between the nickel oxide and the ferrochromium with substantial reduction of the nickel oxide to metallic nickel and substantial oxidation of the carbon of the ferrochromium to an oxide of carbon, and withdrawing the oxide of carbon formed in the reaction from said confined zone, whereby to yield a residue low in carbon and including metallic iron, nickel and chromium.

3. A process for making a three-metal furnace additive for use in making stainless steels containing iron, cobalt and chromium which comprises reacting in the solid state cobalt oxide with ferrochromium containing a substantial amount of carbon by heating an intimate mixture of said cobalt oxide and said ferrochromium in a confined zone under conditions of low absolute pressure to a temperature below the point of substantial fusion of said materials and for a time sufficient to effect reaction between the cobalt oxide and the ferrochromium with substantial reduction of the cobalt oxide to metallic cobalt and substantial oxidation of the carbon of the ferrochromium to an oxide of carbon, and withdrawing the oxide of carbon formed in the reaction from said confined zone, whereby to yield a residue low in carbon and including metallic iron, cobalt and chromium.

4. A process for making a three-metal furnace additive for use in making stainless steels containing iron, nickel and manganese which comprises reacting in the solid state nickel oxide with ferromanganese containing a substantial amount of carbon by heating an intimate mixture of said nickel oxide and said ferromanganese in a confined zone under conditions of low absolute pressure to a temperature below the point of substantial fusion of said materials and for a time sufficient to effect reaction between the nickel oxide and the ferromanganese with substantial reduction of the nickel oxide to metallic nickel and substantial oxidation of the carbon of the ferromanganese to an oxide of carbon, and withdrawing the oxide of carbon formed in the reaction from said confined zone, whereby to yield a residue low in carbon and including metallic iron, nickel and manganese.

5. A process for making a three-metal furnace additive for use in making stainless steels which comprises reacting in the solid state a material containing a substantial proportion of nickel oxide in intimate contact with a ferroalloy material containing a substantial amount of carbon by heating said materials in a confined zone under conditions of low absolute pressure to a temperature below the point of substantial fusion of said materials and for a time sufficient to effect reaction between the nickel oxide and the ferroalloy with substantial reduction of the

nickel oxide to metallic nickel and substantial oxidation of the carbon of the ferroalloy to an oxide of carbon, and withdrawing the oxide of carbon formed in the reaction from said confined zone, whereby to yield a metallic residue low in carbon including nickel and the metals of the ferroalloy in the metallic state.

6. A process for making a three-metal furnace additive for use in making stainless steels which comprises reacting in the solid state a material containing a substantial proportion of a metal oxide selected from the group consisting of nickel oxide and cobalt oxide in intimate contact with a ferroalloy material containing a substantial amount of carbon selected from ferroalloys of the group consisting of ferrochromium, ferromanganese, ferrotitanium, ferromolybdenum, ferrotungsten, ferrovanadium, ferrozirconium, ferrohafnium, and mixtures and composite alloys thereof by heating said materials in a confined zone at a temperature below the point of substantial fusion of said materials of from 1000° C. to 1400° C., at a pressure of about 150 microns of mercury at the end of the reaction period and for a time sufficient to effect reaction between the metal oxide-containing material and the ferroalloy with substantial reduction of the metal of the metal oxide to the metallic state and substantial oxidation of the carbon of the ferroalloy to an oxide of carbon, and withdrawing the oxide of carbon formed in the reaction from said confined zone, whereby to yield a metallic residue low in carbon including the metal of the metal oxide-containing material and the metals of the ferroalloy substantially all in the metallic state.

7. A process for making a three-metal furnace additive for use in making stainless steels containing iron, nickel and chromium which comprises reacting in the solid state nickel oxide with ferrochromium containing a substantial amount of carbon by heating an intimate mixture of said nickel oxide and said ferrochromium in a confined zone at a temperature below the point of substantial fusion of said materials of from 1000° C. to 1400° C., at a pressure of about 150 microns of mercury at the end of the reaction period and for a time sufficient to effect reaction between the nickel oxide and the ferrochromium with substantial reduction of the nickel oxide to metallic nickel and substantial oxidation of the carbon of the ferrochromium to an oxide of carbon, and withdrawing the oxide of carbon formed in the reaction from said confined zone, whereby to yield a residue low in carbon and including metallic iron, nickel and chromium.

8. A ferroalloying furnace additive in the form of finely divided largely unfused particles each comprising a metal of the group consisting of nickel and cobalt and mixtures thereof in amounts ranging from 20% to 50%, a metal of the group consisting of chromium, manganese, titanium, molyddenum, tungsten, vanadium, zirconium, hafnium and mixtures thereof in amounts ranging from 25% to 65%, carbon in amounts ranging from 0.01% to 0.15%, the alloying metal content of said aggregate being greater than 45%, the balance being substantially all iron except for incidental metallic and metallic oxide constituents.

9. A ferroalloying furnace additive in the form of a sintered, porous, dense aggregate of finely divided largely unfused particles each comprising nickel in amounts ranging from 20% to 50%, chromium in amounts ranging from 25% to 65%, carbon in amounts ranging from 0.01% to 0.15%, the balance being substantially all iron except for incidental metallic and metallic oxide constituents.

10. A ferroalloying furnace additive in the form of a sintered, porous, dense aggregate of finely divided largely unfused particles each comprising cobalt in amounts ranging from 20% to 50%, chromium in amounts ranging from 25% to 65%, carbon in amounts ranging from 0.01% to 0.15%, the balance being substantially all iron except for incidental metallic and metallic oxide constituents.

11. A ferroalloying furnace additive in the form of a

sintered, porous, dense aggregate of finely divided largely unfused particles each comprising nickel in amounts ranging from 20% to 50%, manganese in amounts ranging from 25% to 65%, carbon in amounts ranging from 0.01% to 0.15%, the balance being substantially all iron except for incidental metallic and metallic oxide constituents.

12. A ferroalloying furnace additive in the form of finely divided, largely unfused particles each comprising nickel in amounts ranging from 20% to 50%, chromium in amounts ranging from 25% to 65%, carbon in amounts ranging from 0.01% to 0.15%, the balance being substantially all iron except for incidental metallic and metallic oxide constituents.

13. The method of introducing nickel into a steel melt which comprises the steps of reacting nickel oxide with ferrochromium having a substantial percentage of carbon therein to produce an iron-chromium-nickel residue in the solid state, and of adding this residue to molten steel.

14. The method of introducing nickel into a steel melt which comprises the steps of reacting nickel oxide with ferrochromium having a substantial carbon content in a vacuum furnace, the oxygen in the oxide being sufficient to combine with substantially all of the carbon, to produce a low carbon chromium-nickel-iron residue, of agglomerating the residue, and of charging the agglomerate to the steel furnace.

15. The method of adding nickel or cobalt to a steel melt which is to contain at least one other metal in addition to iron in its final specification which comprises the steps of first incorporating the nickel or cobalt as a component of an alloy containing the other metal, and then adding this alloy in its solid state to the steel melt.

16. The method of making a steel containing in addition to iron one metal of a first group of nickel and cobalt, and one or more metals of a second group consisting of chromium, manganese, titanium, molybdenum, tungsten, vanadium, zirconium, and hafnium, which comprises an initial step of reacting the metal or materials of the second group at temperatures close to but below their melting point under comparatively high vacuum conditions with the oxide of the metal of the first group to produce an intermediate product composed of metals of both groups with carbon below 0.2%, and of then adding this product to a steel melt.

17. The method of making a chromium-nickel steel which comprises an initial step of reacting nickel oxide with high carbon ferrochromium at temperatures close to but below their melting point under comparatively high vacuum conditions to produce an intermediate product consisting essentially of metallic iron, chromium and nickel with carbon below 0.2%, and of then adding this intermediate product to a steel melt to produce a chromium-nickel steel of exact metal proportions.

18. A ferroalloying material in the form of a sintered, porous, dense aggregate of finely divided particles comprising metallic chromium, nickel, and iron with negligible metallic oxides and oxide constitutents, the chromium-nickel ratio being 1.5–2.5 of chromium to 1 of nickel.

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