



US 20160168654A1

(19) **United States**(12) **Patent Application Publication**
TSUCHIYA et al.(10) **Pub. No.: US 2016/0168654 A1**(43) **Pub. Date: Jun. 16, 2016**(54) **METHOD FOR MANUFACTURING
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(Kobe Steel, Ltd., Hyogo (JP))**(21) Appl. No.: **14/908,055**(22) PCT Filed: **May 26, 2014**(86) PCT No.: **PCT/JP14/63829**

§ 371 (c)(1),

(2) Date: **Jan. 27, 2016**(30) **Foreign Application Priority Data**

Jul. 25, 2013 (JP) 2013-154793

Publication Classification(51) **Int. Cl.****C21B 13/14** (2006.01)**C22B 1/02** (2006.01)**C22B 1/16** (2006.01)**C21B 13/00** (2006.01)(52) **U.S. Cl.**CPC **C21B 13/146** (2013.01); **C21B 13/0046**
(2013.01); **C21B 13/0066** (2013.01); **C22B**
1/02 (2013.01); **C22B 1/16** (2013.01)(57) **ABSTRACT**

A process for producing an agglomerate comprising heat treating an iron oxide-containing powder at a heating temperature of 900 to 1,200° C., and granulating an obtained heat treated powder, as a raw material, thereby producing an agglomerate, wherein the iron-oxide-containing powder has a 50% particle diameter of 2 µm or less.

FIG. 1

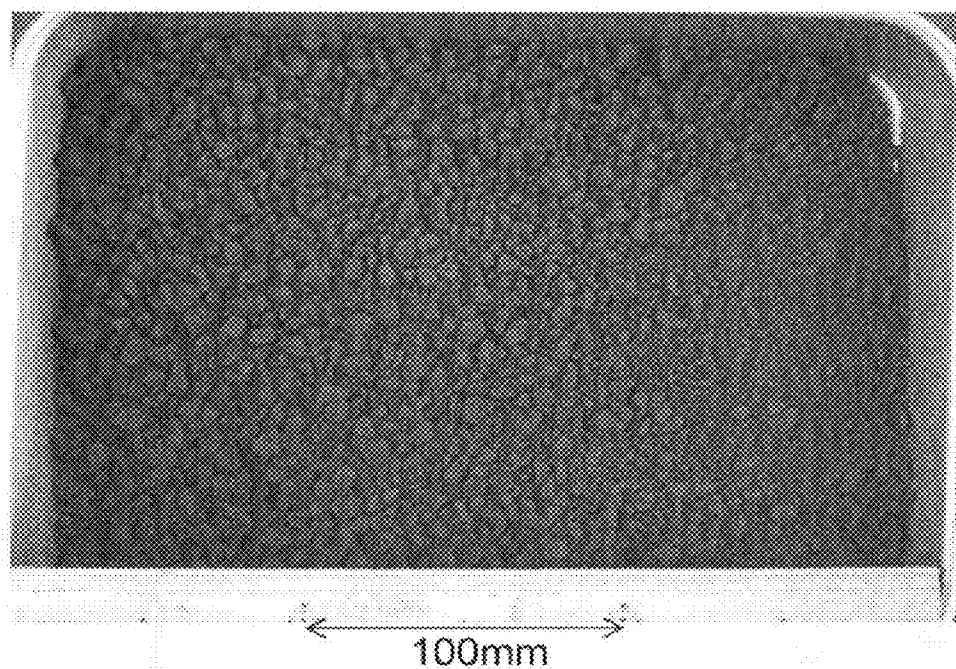
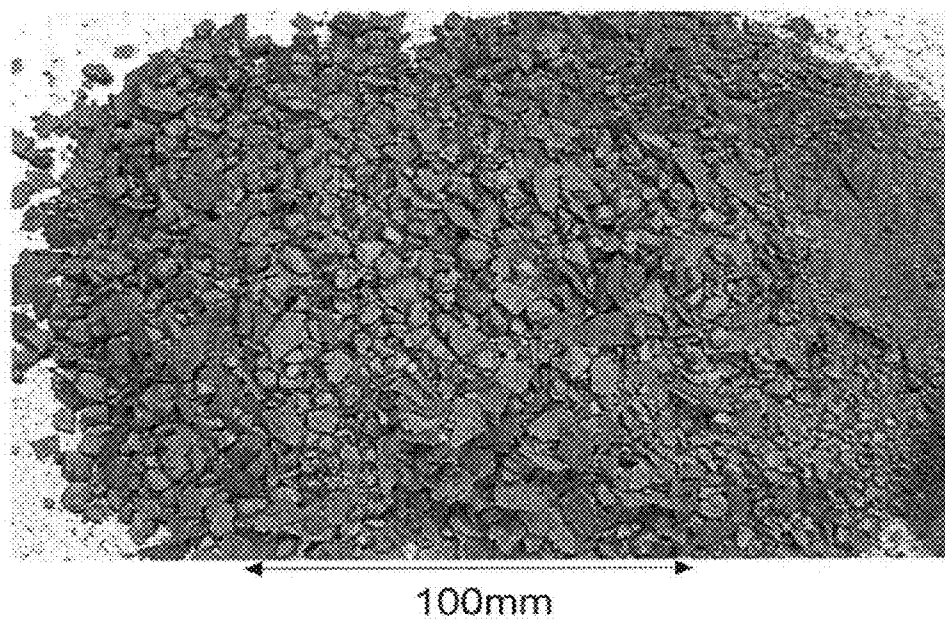


FIG. 2



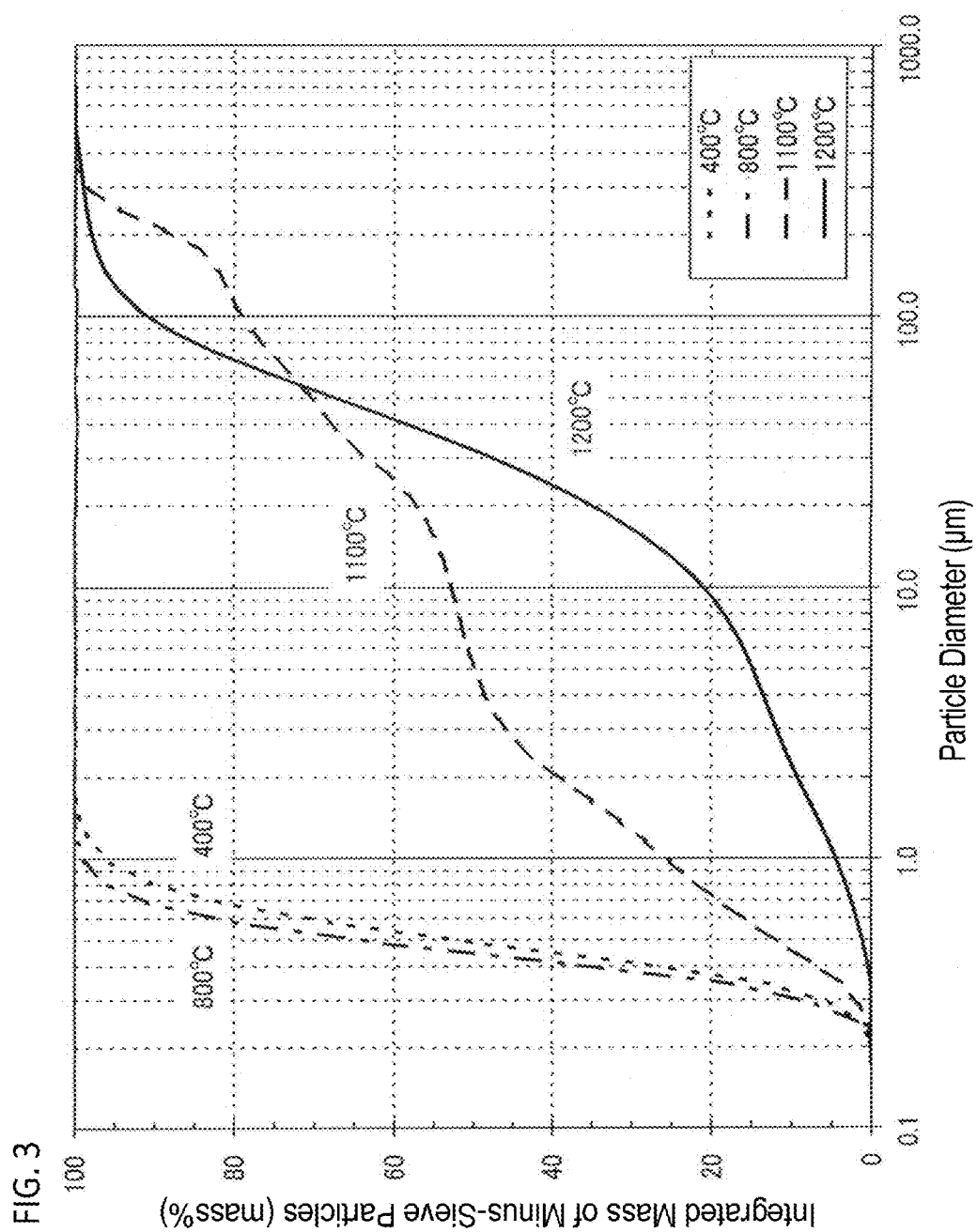


FIG. 4

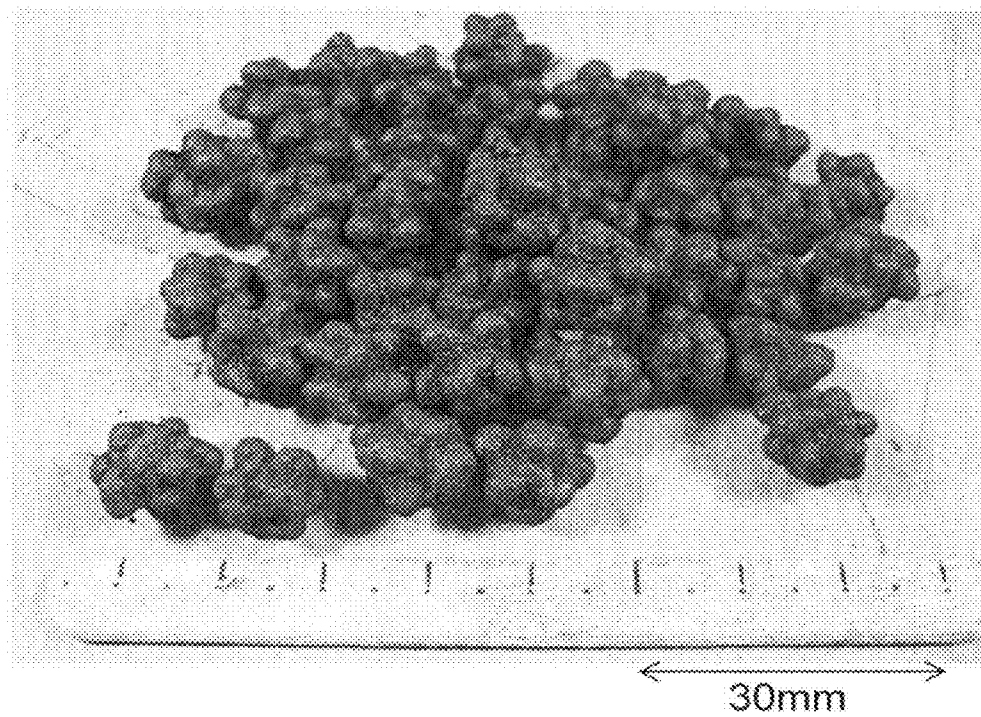
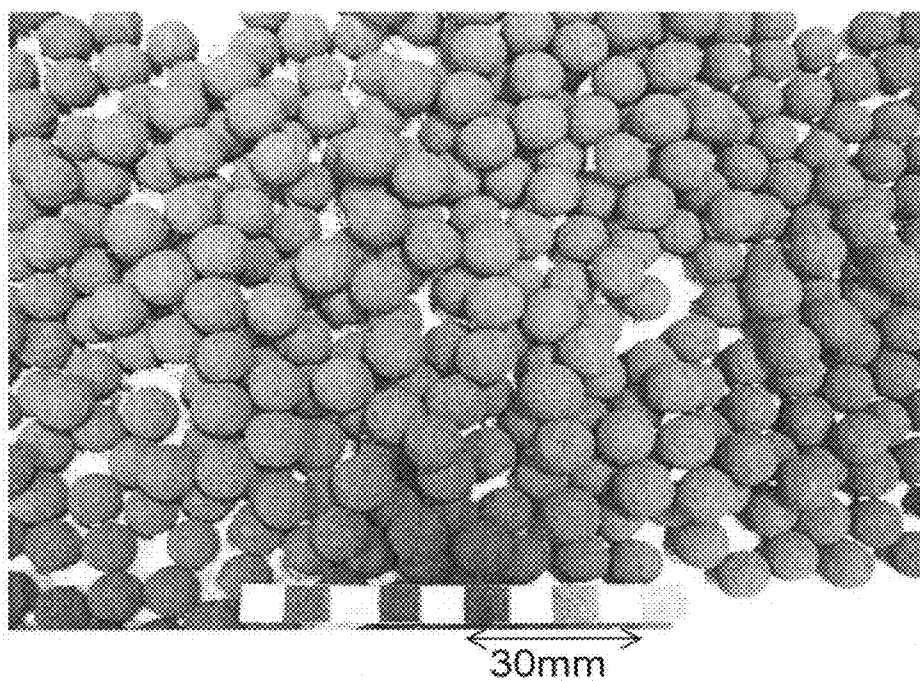


FIG. 5



METHOD FOR MANUFACTURING AGGLOMERATE AND REDUCED IRON

TECHNICAL FIELD

[0001] The present invention relates to a technique for effectively utilizing a fine iron-oxide-containing powder having a 50% particle diameter of 2 μm or less as an iron source.

BACKGROUND ART

[0002] As a process for producing reduced iron from an iron-oxide-containing substance such as an iron ore, for example, a gas reduction method in which natural gas is utilized is known. As production processes of reduced-iron which were developed in recent years, examples thereof include the FASTMET process in which agglomerates obtained by mixing an iron-oxide-containing substance with a carbonaceous reducing agent, e.g., a carbonaceous material, are heated at a high temperature of 1,300° C. or more to produce reduced agglomerates, and the ITmk3 process in which the reduced agglomerates are further heated and melted and the melt is separated into reduced iron and slag to produce granular reduced iron.

[0003] For producing reduced iron from an iron-oxide-containing substance in the manner described above, use is made of agglomerates having a diameter of 13 to 18 mm obtained by mixing the iron-oxide-containing substance as a raw material with water and a binder in a mixer and granulating the mixture with a granulator.

[0004] As methods for agglomerating a powder, for example, a pelletizing method and a sintering method are known. Granulation methods suitable as pretreatments for powder particle size ranges have been prescribed for (for example, Non-Patent Document 1). Specifically, a 50% particle diameter of 4 μm or larger is recommended for the rolling granulation method as one example of the pelletizing method, and a 50% particle diameter of about 0.11 to 3 mm is recommended for the sintering method.

[0005] Meanwhile, examples of valuable metals other than iron include Ni, Al, Ti, etc. These valuable metals are being separated and recovered as Ni, Al, and Ti from Ni-containing ores such as saprolite, Al-containing ores such as red mud, Ti-containing ores such as ilmenite, etc. For example, the high pressure acid leach (HPAL) process is known as a process for separating and recovering Ni from an Ni-containing ore. In this process, Ni can be extracted and recovered by stably reacting an Ni-containing ore with sulfuric acid kept in a high-temperature high-pressure state. After the extraction and recovery of Ni, a product of sedimentation separation is yielded as a residue. This residue contains iron oxides in a large amount, and these oxides are mainly accounted for by hematite (Fe_2O_3). This residue has a water content of 20% or higher, is in a muddy state, and has a 50% particle diameter as small as about 0.6 μm .

PRIOR ART DOCUMENT

Non-Patent Document

[0006] Non-Patent document 1: "Tetsutohagane", Relation between Ore-Grindability and Optimum Size for Pelletizing Nihon Tekko Kyokai-shi, 49th year (1963), No. 3, pp. 346-348

SUMMARY OF THE INVENTION

Problems that the Invention is to Solve

[0007] These are cases where the residues (hereinafter often referred to as tailings) which have remained after desired components were recovered by beneficiation operations contain iron oxides such as hematite in a large amount as stated above. It is hence conceived that the iron oxides contained in the tailings are reduced, i.e., are utilized as an iron source. However, since the tailings usually are exceedingly fine, it is difficult to agglomerate the tailings by the rolling granulation method to obtain granules usable as an ironmaking raw material. The reason for this is as follows. In the case where the particles are exceedingly fine, the particles readily stick to one another during stirring within a mixer to form pseudo-particles. Upon granulation with a granulator, these pseudo-particles bond to one another to grow, thereby forming pellets each having projections on the surface like konpeito. Pellets of such a shape are uneven in internal structure and low in strength and, hence, cannot be used as an ironmaking raw material. It is therefore difficult to effectively utilize the tailings as an iron source by agglomerating the tailings to obtain an ironmaking raw material.

[0008] The present invention has been achieved under such circumstances. An object thereof is to provide a process for producing agglomerates by granulating a fine iron-oxide-containing powder having a 50% particle diameter of 2 μm or less to produce agglomerates usable as an ironmaking raw material. Another object of the present invention is to provide a technique for producing reduced iron from the agglomerates obtained by agglomeration.

Means for Solving the Problems

[0009] The present inventors diligently made investigations in order to agglomerate a fine iron-oxide-containing powder and use the agglomerates as an ironmaking raw material. As a result, the present inventors have found that when an iron-oxide-containing powder having a 50% particle diameter of 2 μm or less is heat-treated at a given temperature, the particles are enlarged through sintering to each other and thus become able to be agglomerated, making it possible to produce agglomerates. The present invention has been thus completed.

[0010] That is, the process for producing an agglomerate which can solve the above problems in the present invention includes: a step of heat-treating an iron-oxide-containing powder having a 50% particle diameter of 2 μm or less at a heating temperature of 900 to 1,200° C., and a step of granulating an obtained heat-treated powder, as a raw material, thereby producing an agglomerate.

[0011] The granulation may be conducted by a rolling granulation method.

[0012] The heat treatment may be conducted so that the heat-treated powder has a 50% particle diameter of 4 μm or larger. For example, the heat treatment may be conducted for a heating period of 30 minutes or longer. The heat treatment is preferably conducted while rolling the iron-oxide-containing powder.

[0013] As the iron-oxide-containing powder, a tailing can be used. As the tailing, for example, a residue which has remained after Ni recovery from a Ni-containing ore can be used.

[0014] In the present invention, a process for producing a reduced iron, in which the agglomerate obtained by the above process is heated, thereby producing a reduced iron, is included. The agglomerate may further contain a carbonaceous reducing agent.

Effects of the Invention

[0015] According to the present invention, by subjecting an iron-oxide-containing powder having a 50% particle diameter of 2 μm or less to a heat treatment at a heating temperature of 900 to 1,200° C., the particles can be enlarged. The resultant particles can be agglomerated by conventional methods, and spherical agglomerates can be produced therefrom. The agglomerates obtained can be utilized as an ironmaking raw material.

BRIEF DESCRIPTION OF THE DRAWINGS

[0016] FIG. 1 is a photograph, as a drawing substitute, of a heat-treated powder obtained by a heat treatment conducted at a heating temperature of 400° C.

[0017] FIG. 2 is a photograph, as a drawing substitute, of a heat-treated powder obtained by a heat treatment conducted at a heating temperature of 1,200° C.

[0018] FIG. 3 is graphs which show the particle size distributions of heat-treated powders.

[0019] FIG. 4 is a photograph, as a drawing substitute, of agglomerates produced from the heat-treated powder obtained by a heat treatment conducted at a heating temperature of 400° C., by disaggregating the heat-treated powder with a ball mill and then granulating the particles.

[0020] FIG. 5 is a photograph, as a drawing substitute, of agglomerates produced from the heat-treated powder obtained by a heat treatment conducted at a heating temperature of 1,200° C., by pulverizing the heat-treated powder with a ball mill and then granulating the particles.

EMBODIMENTS FOR CARRYING OUT THE INVENTION

[0021] The process in the present invention includes

[0022] a step of heat-treating an iron-oxide-containing powder having a 50% particle diameter of 2 μm or less at a heating temperature of 900 to 1,200° C. (hereinafter often referred to as heat treatment step) and

[0023] a step of granulating an obtained heat-treated powder, as a raw material, thereby producing an agglomerate (hereinafter often referred to as agglomeration step). Each step is explained below in detail.

[0024] [Heat Treatment Step]

[0025] In the process in the present invention, it is essential to use an iron-oxide-containing powder having a 50% particle diameter of 2 μm or less. This process is intended to be used for granulating such a fine iron-oxide-containing powder to obtain agglomerates and effectively utilizing the agglomerates as an iron source.

[0026] As the iron-oxide-containing powder having a 50% particle diameter of 2 μm or less, tailings can be used. The term "tailing(s)" means the residue which has remained after desired components were recovered by a beneficiation operation, and the kind of the ore to be beneficiated is not particularly limited. As the tailings, examples thereof include the residue resulting from beneficiation of an iron ore, the residue which has remained after recovery of Al from an Al-containing ore, the residue which has remained after recovery of Ti

from a Ti-containing ore, the residue which has remained after recovery of Ni from an Ni-containing ore, or the like.

[0027] Use is being made of red mud as an Al-containing ore, ilmenite as a Ti-containing ore, saprolite as an Ni-containing ore, or the like. For example, the HPAL process described above is known as a process for recovering Ni from an Ni-containing ore, and the residue which has remained after the separation and recovery of Ni has a 50% particle diameter of 2 μm or less.

[0028] In the heat treatment step, the iron-oxide-containing powder having a 50% particle diameter of 2 μm or less is heat-treated at a heating temperature of 900 to 1,200° C. By heat-treating the fine iron-oxide-containing powder at a temperature within that range, the iron-oxide-containing powder is oxidized and enlarged through sintering. As a result, the particles can be grown to such a size that the enlarged particles can be agglomerated in the step which will be described later. In case where the heating temperature is lower than 900° C., the enlarging effect is not obtained and the resultant particles cannot be agglomerated or can be agglomerated to only give agglomerates which are not spherical. Consequently, the heating temperature is 900° C. or higher, preferably 950° C. or higher, more preferably 1,000° C. or higher. However, in case where the heating temperature exceeds 1,200° C., a problem arises in that coarse agglomerates are formed or agglomerates adhere to the surface of the heat treatment device. Consequently, the heating temperature is 1,200° C. or lower, preferably 1,150° C. or lower, more preferably 1,100° C. or lower.

[0029] The heating temperature may be controlled by inserting a thermocouple into the furnace to measure the temperature of the atmosphere at the center of the furnace and regulating the heating temperature on the basis of the measured temperature.

[0030] In the heat treatment, the heating period may be controlled, while taking account of the heating temperature, so that the resultant heat-treated powder has a 50% particle diameter of 4 μm or larger. It is preferable that the heating period should be, for example, 30 minutes or longer. The heating period is more preferably 40 minutes or longer, even more preferably 50 minutes or longer. There is no particular upper limit on the heating period. However, even when the heating period is prolonged, not only the effect of increasing the particle diameter is not enhanced any more but the productivity decreases. Because of this, the heating period may be, for example, 60 minutes or less.

[0031] The heat treatment may be conducted in an oxidizing atmosphere. For example, the treatment may be conducted in the air.

[0032] It is preferable that the heat treatment should be conducted while rolling the iron-oxide-containing powder, in order to evenly heat the powder. As the heating furnace, a rotary heating furnace may be used. The term "rotary heating furnace" means a furnace in which the furnace surface which is the heating surface is rotating on an axis of rotation and this axis of rotation lies at an angle in the range of from the horizontal to less than the vertical.

[0033] [Agglomeration Step]

[0034] In the agglomeration step, the heat-treated powder obtained in the heat treatment step is used as a raw material and this heat-treated powder is agglomerated to produce agglomerates.

[0035] Examples of methods for granulating the heat-treated powder include a rolling granulation method.

[0036] It is preferred to agglomerate the heat-treated powder so that the agglomerates have a particle diameter of, for example, 10 to 16 mm.

[0037] Prior to the granulation, the heat-treated powder may be disaggregated or pulverized. As a disaggregating machine or pulverizer, a known one can be used. For example, use can be made of a ball mill, roller mill, roll crusher or the like.

[0038] [Others]

[0039] The agglomerates obtained in the agglomeration step can be used as an ironmaking raw material. For example, the agglomerates obtained are subjected to a thermal hardening treatment and then introduced into a blast furnace. Alternatively, the thermally hardened agglomerates obtained by the thermal hardening treatment are further heated in a reducing gas atmosphere. Thus, the iron oxide can be reduced to produce reduced iron.

[0040] Reduced iron can be produced also by further adding a carbonaceous reducing agent, a binder, etc. to the heat-treated powder, forming the mixture into agglomerates, and heating the agglomerates in a heating furnace.

[0041] As described above, according to the present invention, an iron-oxide-containing powder having a 50% particle diameter of 2 μm or less can be enlarged to a particle diameter which renders granulation possible, by heat-treating the powder at a temperature within a given range. Consequently, when the heat-treated powder obtained by the heat treatment is agglomerated as a raw material, the particles of the heat-treated powder grow at a rapidly accelerating rate and agglomerates having an even structure can be produced.

[0042] This application claims a right of priority based on Japanese Patent Application No. 2013-154793 filed on Jul. 25, 2013. The entire contents of the description of Japanese Patent Application No. 2013-154793 are incorporated herein by reference.

[0043] The present invention will be explained below in more detail by reference to Example. However, the present invention should not be construed as being limited by the following Example, and can of course be modified so long as the modifications do not depart from the spirit which was described above or will be described later. Such modifications are all included in the technical range of the present invention.

Example

[0044] An iron-oxide-containing powder having a 50% particle diameter of 2 μm or less was heat-treated, and the heat-treated powder obtained was agglomerated to produce agglomerates. A detailed explanation thereof is given below.

[0045] As the iron-oxide-containing powder having a 50% particle diameter of 2 μm or less, use was made of a residue which had remained after Ni recovery from an Ni-containing ore. This residue was tailings and had a water content of about 27%. The component composition of the residue which had remained after Ni recovery is shown in Table 1 below. In Table 1, LOI means ignition loss.

[0046] The tailings were placed outdoors and exposed to sunlight to reduce the water content to about 19%. The tailings having a water content regulated to about 19% were reddish brown. A 2 kg portion thereof was introduced into a rotary heating furnace. The tailings were heat-treated, while being allowed to roll, and were dried and sintered thereby. For the heat treatment, a heating temperature of 400° C., 800° C., 1,100° C., or 1,200° C. was used as shown in Table 2. The heating period was about 60 minutes in the case where the

heating temperature was 400° C., and was about 30 minutes in the case where the heating temperature was 800° C., 1,100° C., or 1,200° C., as shown in Table 2. With respect to the heating atmosphere, the heat treatment was conducted in an air stream.

[0047] The powder obtained through the heat treatment remained reddish brown in the case where the heating temperature was 400° C., 800° C., or 1,100° C. However, the powder changed to blackish brown in the case where the heating temperature was 1,200° C. For reference, a photograph of the powder obtained through the heat treatment at a heating temperature of 400° C. is shown as a drawing substitute in FIG. 1. A photograph of the powder obtained through the heat treatment at a heating temperature of 1,200° C. is shown as a drawing substitute in FIG. 2.

[0048] Next, after the heat treatment, each heat-treated powder cooled to room temperature was disaggregated or pulverized with a ball mill to obtain a sample to be agglomerated. With respect to the heat-treated powders obtained through the heat treatment at a heating temperature of 400° C., 800° C., or 1,100° C., each powder was disaggregated with a ball mill for about 30 seconds. Meanwhile, the heat-treated powder obtained through the heat treatment at a heating temperature of 1,200° C. was pulverized with a ball mill for about 20 minutes.

[0049] The particle size distribution of each heat-treated powder was measured, and the results thereof are shown in FIG. 3. The abscissa of FIG. 3 indicates particle diameter (μm), while the ordinate thereof indicates the integrated mass of minus-sieve particles (mass %). As shown in FIG. 3, when the particle size distribution of a heat-treated powder is determined, with the mass of the whole powder being taken as 100%, the particle diameter corresponding to the point where the integrated mass of minus-sieve particles reaches 50% is called 50% particle diameter.

[0050] The 50% particle diameter (μm), the integrated mass of minus-sieve particles having a particle diameter of less than 1 μm (mass %), and the integrated mass of minus-sieve particles having a particle diameter of less than 10 μm (mass %) were each calculated, and the results thereof are shown in Table 2 below.

[0051] As apparent from FIG. 3 and Table 2, the powders obtained through the heat treatment conducted at a heating temperature of 400° C. or 800° C. each had substantially the same 50% particle diameter as the raw material powder which had not been heat-treated, and the integrated mass of minus-sieve particles having a particle diameter of less than 1 μm thereof was also substantially the same as that of the raw material powder. It was thus found that the raw material powder and the powders obtained through the heat treatment conducted at a heating temperature of 400° C. or 800° C. each had a particle diameter of less than 10 μm and had substantially the same particle size configuration. In contrast, the powder obtained through the heat treatment conducted at a heating temperature of 1,100° C. had a 50% particle diameter which was about 8.6 times that of the raw material powder which had not been heat-treated, showing that the particles have enlarged due to the heat treatment. Meanwhile, the powder obtained through the heat treatment conducted at a heating temperature of 1,200° C. had a 50% particle diameter which was about 53.5 times that of the raw material powder which had not been heat-treated, and the integrated mass of minus-sieve particles having a particle diameter of less than 1

um was able to be reduced to 4.4 mass %. It can be found that the particles have enlarged due to the heat treatment.

[0052] The enlargement of particles can be seen from not only the results concerning the integrated mass of minus-sieve particles having a particle diameter of less than 1 μm but also the results concerning the integrated mass of minus-sieve particles having a particle diameter of less than 10 μm . Namely, the raw material powder and the powders obtained through the heat treatment conducted at a heating temperature of 400° C. or 800° C. each were composed only of particles having a particle diameter of less than 10 μm , whereas the heat treatment conducted at a heating temperature of 1,200° C. was able to reduce the proportion of particles having a particle diameter of less than 10 μm to 20.9% and to increase the proportion of coarse particles having a particle diameter of 10 μm or larger to about 80%.

[0053] Next, the specific surface area (cm^2/g) of each heat-treated powder was determined by calculation on the basis of the particle size distribution values thereof for respective particle size ranges on the assumption that each particle diameter was spherical. The results thereof are shown in Table 2 given later.

[0054] As apparent from Table 2, the powders obtained through the heat treatment conducted at a heating temperature of 400° C. or 800° C. each had a specific surface area (calculated value) of 27,400 to 29,380 cm^2/g . In contrast, the powder obtained through the heat treatment conducted at a heating temperature of 1,100° C. had a specific surface area (calculated value) of 8,520 cm^2/g , and the powder obtained through the heat treatment conducted at a heating temperature of 1,200° C. had a specific surface area (calculated value) of 1,920 cm^2/g . It can be found from these results that as the heating temperature was elevated, the specific surface area became smaller and the particles became larger.

[0055] Next, each heat-treated powder was introduced into a granulator made of a rubber tire having a diameter of about 35 cm, and an appropriate amount of water was added thereto to conduct granulation. As a result, in the case where a powder obtained by disaggregating the heat-treated powder obtained through the heat treatment at a heating temperature of 400° C. or 800° C. was used, the resultant pellets did not have a spherical shape and had surface projections like konpeito. A photograph of the agglomerates produced by granulating the powder obtained by disaggregating the heat-treated powder obtained through the heat treatment at a heating temperature of 400° C. is shown as a drawing substitute in FIG. 4.

[0056] In contrast, in the case where a powder obtained by pulverizing the heat-treated powder obtained through the heat treatment at a heating temperature of 1,100° C. or 1,200° C. was used, the resultant pellets had a spherical shape. A pho-

tograph of the pellets produced by granulating the powder obtained by pulverizing the heat-treated powder obtained through the heat treatment at a heating temperature of 1,200° C. is shown as a drawing substitute in FIG. 5.

[0057] Next, the pellets obtained by granulating the heat-treated powder obtained through the heat treatment conducted at a heating temperature of 1,100° C. or 1,200° C. were examined for water content (%), crushing strength (kg) per pellet, and porosity (%).

[0058] The crushing strength was determined by placing one pellet between two flat plates, applying a load to the flat plates so as to compress the pellet, and measuring the load at the time when the pellet fractured (hereinafter the load is also called crushing load; unit, kg), with a strength tester. The measurement of crushing load was made on ten pellets, and an average thereof was determined. The results thereof are shown in Table 2.

[0059] The porosity (%) was determined through calculation from the value of apparent specific gravity, which was determined on the basis of the buoyancy of a pellet immersed in mercury, and from the value of true specific gravity of the raw material powder mixed. The results thereof are shown in Table 2.

[0060] It was able to be ascertained that in the case where the heat-treated powder obtained through the heat treatment conducted at a heating temperature of 1,100° C. or 1,200° C. was agglomerated, the pellets obtained had substantially the same water content, crushing strength, and porosity as the green pellets produced in conventional pelletizing plants.

[0061] By subjecting these pellets to a thermal hardening treatment and then heating the pellets, for example, in a reducing gas atmosphere, reduced iron can be produced. Reduced iron can be produced also by adding a carbonaceous reducing agent, a binder, etc. to the heat-treated powder to prepare pellets and heating the pellets.

[0062] As described above, according to the present invention, an iron-oxide-containing powder having a 50% particle diameter of 2 μm or less can be made to have a particle size which renders granulation possible, by heat-treating the powder at a heating temperature of 900 to 1,200° C., and agglomerates can be produced therefrom. These agglomerates can be effectively utilized as an iron source.

TABLE 1

Component composition (mass %)								
T.Fe	FeO	CaO	SiO ₂	Al ₂ O ₃	S	Cr	Ni	LOI
62.02	0.06	0.01	2.65	0.62	1.05	1.46	0.02	4.82

TABLE 2

No.	Heat	Heat	50%	Smaller	Smaller	Specific	Properties of wet pellets			
	treatment temperature (° C.)	treatment period (min)	particle diameter (μm)	than 1 μm (mass %)	than 10 μm (mass %)	surface area (cm2/g)	Shape of agglomerates	Water content (%)	Crushing strength (kg)	Porosity (%)
1	raw material	—	0.6	98.8	100	—	—	—	—	—
2	400	60	0.5	95.7	100	27400	konpeito shape	—	—	—
3	800	30	0.4	98.8	100	29380	konpeito shape	—	—	—

TABLE 2-continued

No.	Heat	Heat	50%	Smaller	Smaller	Specific	Shape of agglomerates	Properties of wet pellets		
	treatment temperature (° C.)	treatment period (min)	particle diameter (μm)	than 1 μm (mass %)	than 10 μm (mass %)	surface area (cm2/g)		Water content (%)	Crushing strength (kg)	Porosity (%)
4	1100	30	5.2	25.6	52.1	8520	spherical	16.5	2.0	38.4
5	1200	30	32.1	4.4	20.9	1920	spherical	10.2	2.5	32.8

1. A process for producing an agglomerate, comprising heat-treating an iron-oxide-containing powder having a 50% particle diameter of 2 μ m or less at a heating temperature of 900 to 1,200° C., and granulating an obtained heat-treated powder, as a raw material, thereby producing an agglomerate.
2. The process according to claim 1, wherein the granulation is conducted by a rolling granulation method.
3. The process according to claim 1, wherein the heat treatment is conducted so that the heat-treated powder has a 50% particle diameter of 4 μ m or larger.
4. The process according to claim 1, wherein the heat treatment is conducted for a heating period of 30 minutes or longer.
5. The process according to claim 1, wherein the heat treatment is conducted while rolling the iron-oxide-containing powder.
6. The process according to claim 1, wherein the iron-oxide-containing powder is a tailing.
7. The process according to claim 6, wherein the tailing is a residue which has remained after Ni recovery from a Ni-containing ore.

8. A process for producing a reduced iron, wherein the agglomerate obtained by the process according to claim 1 is heated, thereby producing a reduced iron.
9. The process according to claim 8, wherein the agglomerate further contains a carbonaceous reducing agent.
10. The process according to claim 2, wherein the heat treatment is conducted so that the heat-treated powder has a 50% particle diameter of 4 μ m or larger.
11. The process according to claim 2, wherein the heat treatment is conducted for a heating period of 30 minutes or longer.
12. The process according to claim 2, wherein the heat treatment is conducted while rolling the iron-oxide-containing powder.
13. The process according to claim 2, wherein the iron-oxide-containing powder is a tailing.
14. The process according to claim 13, wherein the tailing is a residue which has remained after Ni recovery from a Ni-containing ore.
15. A process for producing a reduced iron, wherein the agglomerate obtained by the process according to claim 2 is heated, thereby producing a reduced iron.

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