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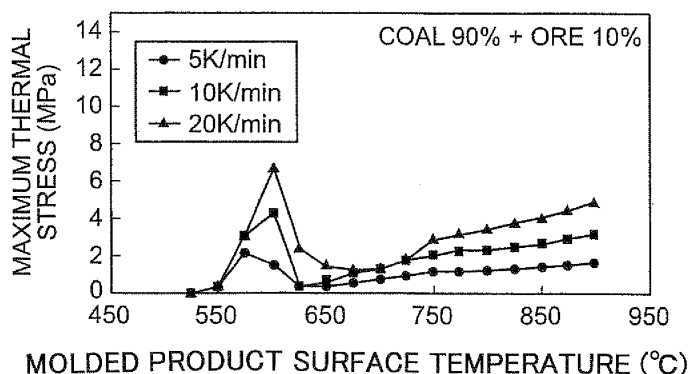
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(54) **METHOD OF PRODUCING FERRO-COKE**

(57) The present invention provides a method of producing ferrocoke capable of, in carbonizing a molded product composed of a carbon-containing material and an iron oxide-containing material to give the ferrocoke, preventing cracking and thermal cracking occurring in carbonizing of the molded product, increasing the original form ratio at the carbonizing furnace discharge side, preventing cracking in charging the ferrocoke into the fur-

nace and also preventing reduction of a yield. Specifically, used is the method for producing ferrocoke by heating a molded product composed of a mixture of a carbon-containing material and an iron oxide-containing material to carbonize the molded product, in which carbonization is performed at a heating rate of 20 °C/min or less in a temperature range where a temperature of a surface of the molded product ranges from 550 °C to 650 °C.

FIG.4



Description**TECHNICAL FIELD**

5 **[0001]** The present invention relates to a method of producing ferrocoke suitable for blast furnace feed by carbonization with use of raw materials of iron ore and coal.

BACKGROUND ART

10 **[0002]** As a method of producing ferrocoke by mixing fine iron ore into raw material coal and carbonizing the mixture in a conventional chamber oven, there have been considered 1) a method of charging fine mixture of coal and fine iron ore into a chamber oven and 2) a method of cold-molding coal and iron ore, that is, forming them at ambient temperatures and charging the molded product into a chamber oven (for example, see "COKE TECHNICAL REPORT", The Fuel Society of Japan, 1958, p.38). However, as the conventional chamber oven is built in silica brick, the iron ore charged
15 therein reacts with silica which is a main ingredient of the silica brick to generate low-melting fayalite (2FeO SiO_2), which gives damages to the silica brick. Hence, the method of producing ferrocoke in the chamber oven has not been performed industrially.

20 **[0003]** Recently, a continuous formed-coke producing method has been developed as a method of producing coke to be replaced with the coke producing method in chamber oven. In the continuous formed-coke producing method, a vertical shaft brick furnace build in chamotte brick not silica brick is used as carbonization oven. After coal is cold-molded into a predetermined size, it is charged into a shaft furnace and heated with use of circulating gas for heat carrier to carbonize a formed coal, and thereby a formed coke is produced. Although it has been already confirmed that coke of strength equal to that produced in the conventional chamber oven can be produced by using a large amount of non-
25 slightly caking coal that is inexpensive and rich in resource reserves, if the used coal is of high caking property, the formed coal is softened and fused in the shaft furnace, which makes the operation in the shaft furnace difficult and brings about degradation of coke such as deformation, crack and the like.

30 **[0004]** In order to prevent fusion in the shaft furnace in the continuous coke producing method, there has been proposed a method of charging iron ore into coal so that the iron ore becomes 15 to 40 % of the total amount, cold-molding into a molded product and charging it into the shaft furnace (see Japanese Patent Application Laid-Open No. 6-55579). In this method, as the iron ore is of less caking property, there is a need to add expensive binder in order to produce the molded product in the cold state. Then, there is also proposed a method of heating and hot-molding coal and iron ore or iron material into block-shaped product (see, for example, Japanese Patent Application Laid-Open Nos. 2004-217914 and 2005-53982). However, in the methods disclosed in the above-mentioned Publication Nos. 6-65579, 2004-217914 and 2005-53982, there remain problems of degradation of coke, such as deformation, crack and the like of the molded
35 product in carbonization as the thermal behavior in carbonization is different between coal and iron ore or iron material.

40 **[0005]** Meanwhile, as to production of the formed coke using only coal as main raw material, consideration has been given to a heating pattern in carbonization of the molded product, or formed coke, to prevent degradation of the coke such as deformation, crack and the like in carbonization and a method has been proposed of designing an optimal heating rate in accordance with the temperature of the molded product (see, for example, Japanese Patent Application Laid-Open Nos. 52-23103 and 7-102260).

45 **[0006]** As described above, in the method of producing ferrocoke using raw materials of coal and iron ore or iron material, the problems of deformation, crack and the like of the molded product in carbonization have not been solved. As the ferrocoke is a mixture of coal (hereinafter referred to as "carbon-containing material") and iron ore or iron material (hereinafter referred to as "iron oxide-containing material"), the thermal and mechanical property in heating is greatly different from that in producing of formed coke, and it is predicted deformation and crack behavior of the molded product in carbonization is different.

50 **[0007]** The present invention was carried out in view of the above-mentioned problems and has an object to provide a method of producing ferrocoke by carbonization of a molded product composed of iron oxide-containing material and carbon-containing material, which method is capable of preventing thermal crack, crack that may occur in carbonization of the molded product, improving the original form ratio at the carbonization discharge side and preventing the ferrocoke from being cracked when being charged into the furnace thereby to prevent reduction of the yield.

DISCLOSURE OF THE INVENTION

55 **[0008]** In order to solve these problems, the present invention provides a method for producing ferrocoke by heating a molded product composed of a mixture of a carbon-containing material and an iron oxide-containing material to carbonize the molded product, wherein carbonization is performed at a heating rate of 20 °C/min or less in a temperature range where a temperature of a surface of the molded product ranges from 550 °C to 650 °C.

[0009] According to the present invention, in carbonizing of a molded product composed of a mixture of the iron oxide-containing material and carbon-containing material, it becomes possible to prevent occurrence of thermal stress inside the molded product, reduction of yield in producing of the ferrocoke and crack in the furnace or before being charged into the furnace.

[0010] Here, the heating rate referred to in the present invention is an instantaneous heating rate (temperature gradient (dT/dt) of the heating pattern), not an average heating temperature ($\Delta T/t$) obtained by dividing an increased temperature ΔT °C by a time t required for temperature rising.

BRIEF DESCRIPTION OF THE DRAWINGS

[0011]

Fig. 1 is a graph illustrating the relation between the maximum thermal stress and the surface temperatures of a coal molded product;

Fig. 2 is a graph illustrating the relation between the maximum thermal stress and the surface temperatures of a molded product of 90% coal and 10% iron ore;

Fig. 3 is a graph illustrating heating rate dependency of the maximum thermal stress and the surface temperatures of the coal molded product;

Fig. 4 is a graph illustrating heating rate dependency of the maximum thermal stress and the surface temperatures of the molded product of 90% coal and 10% iron ore; and

Fig. 5 is a graph illustrating the relation between the contraction coefficient and temperature of a mixture of coal and iron ore and of 100% coal.

BEST MODES FOR CARRYING OUT THE INVENTION

[0012] The inventors of the present invention have studied the method of producing ferrocoke, made measurement of the thermal and mechanical property of a molded product of mixed iron oxide-containing material and carbon-containing material as raw materials of ferrocoke, performed thermal stress analysis based on this property and found the heating method optimal for control cracking of the molded product of mixed iron oxide-containing material and carbon-containing material based on analysis results of deformation and cracking of the molded product in heat treatment under various conditions thereby completing the present invention. Here, in the present invention, the iron oxide-containing material is a material such as iron ore containing as main component Fe_2O_3 or Fe_3O_4 , reduced iron containing iron oxide, and iron containing sludge. Besides, the carbon-containing material is a material such as coal, bituminous material and oil coke. The coal includes coal for coke making that shows caking property, coal for general use, such as semianthracite, anthracite and bituminous coal, that does not show caking property, and solvent refined coal such as swelling coal and SRC. The bituminous material includes coal such as pitch, soft pitch, middle softening point pitch, and hard pitch and petroleum bituminous material such as ASP (asphalt pitch) and PDA (propane deasphalting asphalt). The oil coke includes fluid coke and delayed coke.

[0013] As an example of various analysis results, the transition of maximum thermal stress occurring inside the molded product when the material is heated is illustrated in Figs. 1 and 2. There are prepared a raw material containing 100 mass% coal as carbon-containing material and a raw material containing 10 mass% iron ore as iron oxide-containing material and 90 mass% coal as carbon-containing material, which are formed into molded products of volumes 18 cc, 50 cc and 92 cc. Then, the molded products are heated at 5K (Kelvin)/min (5 °C/min) of constant temperature gradient. The transition of maximum thermal stress caused inside the molded product containing 100 mass% coal is illustrated in Fig. 1 and the transition of maximum thermal stress caused inside the molded product containing mixture of 10 mass% iron ore and 90 mass% coal is illustrated in Fig. 2. Here, as to the thermal stress, first, the density, thermal conductivity, specific heat, Young's modulus and temperature-dependent contraction coefficient of the coal and iron oxide-containing coal are measured and this measurement data is used as a basis to calculate the temperature dependency of the thermal stress by heat transfer and thermal stress calculation. The contraction coefficient is measured by a thermomechanical analyzer, using a test sample of a cylindrically molded product of diameter 5 mm and height 8 mm obtained by mixing predetermined amounts of coal and iron ore. The Young's modulus is measured by the resonance method, using a test sample obtained by mixing predetermined amounts of coal and iron oxide and cutting the mixture into a platy of width 15 mm, length 80 mm and thickness 10 mm. The Poisson's ratio is measured based on the method disclosed in the publication (J.Fukai, T.Hashida, K.Suzuki, T.Miura and S.Ohtani: Tetsu-to-Hagané, vol.74 (1988), p.2209) and the specific heat, thermal conductivity and density are measured based on the methods disclosed in the publication (K.Matsubara, O.Tajima, N.Suzuki, Y.Okada, Y.Nakayama and T.Kato: Tetsu-to-Hagané, vol.68 (1982), p.2148). Here, the above-mentioned heat transfer and thermal stress calculation uses the method based on the publication (T.Miura, H.Yoshino, S.Saito, S.Otani: Journal of the Fuel Society of Japan, vol.68 (1989), p.1045)

[0014] As illustrated in Fig. 1, when a molded product composed of only carbon-containing material is carbonized, a of maximum thermal stress is found around 700 °C to 750 °C of the surface temperature of the molded product. Reason for this result is explained with reference to Fig. 5.

[0015] Fig. 5 is a graph illustrating temperature dependency of contraction coefficient of a molded product composed of 100 mass% coal, a molded product composed of 90 mass% coal and 10 mass% iron ore and a molded product composed of 70 mass% coal and 30 mass% ore. As illustrated in Fig. 5, in the heat treatment process of the coal 100 mass% material, the peak (that is, secondary peak of the contraction coefficient) is observed around 750 °C. In this way, the contractile rate of the surface becomes maximum when the surface temperature is around 750 °C, while as the temperature inside the material is lower than that of the surface, the contractile rate is relatively low as compared with that of the surface and there is higher possibility that cracking occurs due to contraction difference between the surface and inside of the molded product. Likewise, the secondary peak is shown of the mixed molded product of coal and iron ore. Here, though the peak (that is, primary peak of the contraction coefficient) is also observed around 500 °C, as the Young's modulus of the coke as molded product composed of 100 mass% coal is small in this primary peak temperature range, as illustrated in Fig. 1, the occurring thermal stress is relatively small and becomes insignificant.

[0016] Besides, as illustrated in Fig. 1, as the molded product volume is larger, the temperature difference between the surface and the inside of the molded product is larger and therefore, the peak value of the maximum thermal stress becomes large.

[0017] Meanwhile, when the molded product composed of a mixture of the carbon-containing material and iron oxide-containing material is carbonized, the contraction coefficient in Fig. 5 is shown. For example, as the thermal conductivity of the iron oxide-containing material is 100 times greater than that of the carbon-containing material, the temperature difference between the surface and inside of the molded product becomes smaller than that of the carbon-containing material only. The same tendency is shown even when the volume of the iron oxide-containing material is increased. Therefore, as illustrated in Fig. 2, the peak of the thermal stress of the mixed molded product of the carbon-containing material and iron oxide-containing material becomes smaller to a degree that it can be ignored around the temperatures of 700 °C to 750 °C. On the other hand, the high peak of thermal stress is shown at the surface temperatures of the molded product ranging from 550 °C to 650 °C where the contraction coefficient ranges from its primary peak value to its minimum value. In the case of the only carbon-containing material (coke of 100 mass% coal), the Young's modulus is small in this temperature range and therefore, the thermal stress becomes insignificant. In the case of the molded product (ferrocake) composed of a mixture of carbon-containing material and iron oxide-containing material, as the Young's modulus is larger due to influence of the iron oxide-containing material, larger thermal stress occurs even with slight change in strain in this temperature range. If the size of the molded product is made smaller to reduce temperature distribution inside the material, Young's modulus dependency is much larger and therefore, the dependency on the molded product volume becomes smaller. In addition, as the bond strength between particles made of carbon-containing material is low in this temperature range, slight increase of occurring thermal stress has great influence on occurrence of crack.

[0018] Thus, the mixture of the carbon-containing material and iron oxide-containing material shows the temperature of the thermal stress which is different from that of the only carbon-containing material. Therefore, as a new finding, in order to prevent occurrence of thermal stress and crack in carbonization, there is only need to control the heating method at the temperatures of 550 °C to 650 °C irrespective of the volume of the molded product, thereby completing the following invention.

[0019] Here, the following heating method at the temperatures of 550 °C to 650 °C used in the present invention is effective in carbonization of a molded product composed of a mixture of the carbon-containing material and iron oxide-containing material obtained by hot briquetting as well as by cold briquetting in which the content of the iron oxide-containing material is increased and a binder is used.

[0020] As the method of controlling the heating at the temperatures of 500 °C to 650 °C, there is a method of controlling the heating rate. As the lower the heating rate, the smaller a temperature difference becomes between the surface and inside of the molded product, thereby to be able to prevent occurrence of the thermal stress. However, when the heating rate is decreased, carbonization time becomes longer, and unpreferably the product productivity is reduced. Then, it is necessary to set the upper limit of the heating rate. Here, the heating rate mentioned in the present invention is not a heating temperature ($\Delta T/t$) obtained by dividing an increased temperature ΔT °C by a time t required in temperature increase, but an instantaneous heating rate (temperature gradient of the heating pattern (dT/dt)).

[0021] For example, the transition of the maximum thermal stress occurring inside the molded product composed of 100 mass% coal as carbon-containing material is illustrated in Fig. 3 and that of the molded product composed of 10 mass% iron ore as iron oxide-containing material and 90 mass% coal as carbon-containing material is illustrated in Fig. 4. Figs. 3 and 4 are graphs each illustrating the transition of the maximum thermal stress occurring inside the molded product when the 18 cc molded product is heated at the heating rates of 5, 10 and 20 K/min (°C/min) of constant temperature gradient. For each raw material, the smaller the heating rate, the smaller the maximum thermal stress.

[0022] As a result of analysis of the cracking and deformation of the molded products that are subjected to heat

treatment under various conditions, it is found that the upper limit of the heating rate at the temperatures of 550 °C to 650 °C in carbonization of the ferrocoke is 20 °C/min (K/min) and when it is heated at the heating rate of 20 °C/min or less that is instantaneous temperature gradient, there occurs almost no crack in the molded product.

[0023] Here, the mass% of the iron oxide-containing material as raw material of the preferable ferrocoke molded product of the present invention is 10 to 30 mass% and the rest is carbon-containing material. The volume of the preferable ferrocoke molded product of the present invention is 6 cc or more.

EXAMPLE 1

[0024] In order to clarify the relation between cracking of the ferrocoke and the ferrocoke thermal treatment conditions, the heating test of the ferrocoke molded product was performed using an electric furnace of which the heating rate is controllable and crack occurrence was investigated.

[0025] First, ferrocoke raw materials were adjusted. The volatile portion, 35 mass% coal (coking coal) as carbon-containing material and iron ore containing 68 mass% Fe as iron oxide-containing material were selected and two raw materials of mixed coal and iron ore were prepared one having a mass ratio of 9:1 and the other having a mass ratio of 7:3. Next, a double-roll type molding machine was used and three molded products were prepared with volumes of 6 cc, 18 cc and 50 cc. These molded products were heated in the electric furnace with various heating patterns.

[0026] A few of molded products formed as mentioned above were arranged in the soaking area of the electric furnace, heated up to 900 °C with various heating patterns under the nitrogen atmosphere, cooled slowly under the nitrogen atmosphere to the ambient temperature and then, taken out of the electric furnace. Then, the appearance of molded products was checked and the ratio of ferrocoke still having its original form (original form ratio) was measured. The ferrocoke having its original form is ferrocoke obtained with no crack in the surface thereof.

[0027] Table 1 shows results of original form ratio obtained when the molded products composed of coal and iron ore at a mass ratio of 7:3 are treated at the heating rate of constant temperature gradient in the temperature ranges of from 550 °C to 650 °C. The temperature rate outside the temperature range of 550 °C to 650 °C is changed appropriately and each temperature rate value is not constant. Here, also for the molded products having raw materials of coal and iron ore at a mass ratio of 9:1, almost the same results were obtained as those of the molded product composed of, as raw material, coal and iron ore at a mass ratio of 7:3 and description of the results of the molded products composed of coal and iron ore at a mass ratio of 7:3 is omitted here.

[0028] For molded products of all volumes and composed of coal and iron ore at both of mass ratios 9:1 and 7:3, no crack was observed at the heating rate 10 °C/min or less at temperatures of 550 °C to 650 °C. In addition, cracked molded products at the heating rate 20 °C/min or less are less than 10% and so few that they do not have influence on the productivity. Meanwhile, when they were heated at the heating rate of 25 °C/min or more exceeding 20 °C/min, it was sure that many cracked ferrocoke were observed.

TABLE 1

Heating rate in 550-650 °C	original form ratio of 6cc molded product	original form ratio of 18cc molded product	original form ratio of 50cc molded product
5 °C/min	0%	0%	0%
10 °C/min	0%	0%	0%
15 °C/min	1%	2%	3%
20 °C/min	3%	4%	7%
25 °C/min	18%	22%	25%
30 °C/min	36%	40%	46%

Claims

1. A method for producing ferrocoke by heating a molded product composed of a mixture of a carbon-containing material and an iron oxide-containing material to carbonize the molded product, wherein carbonization is performed at a heating rate of 20 °C/min or less in a temperature range where a temperature of a surface of the molded product ranges from 550 °C to 650 °C.

FIG.1

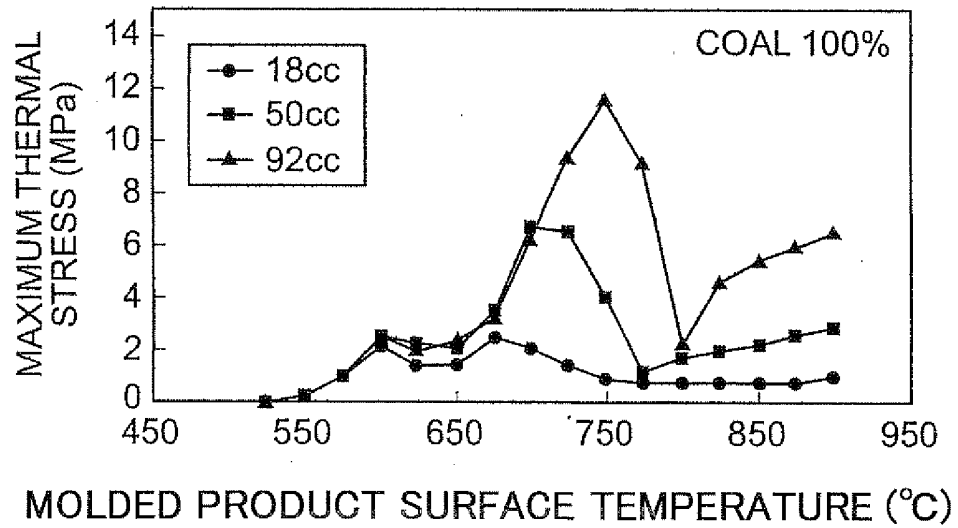


FIG.2

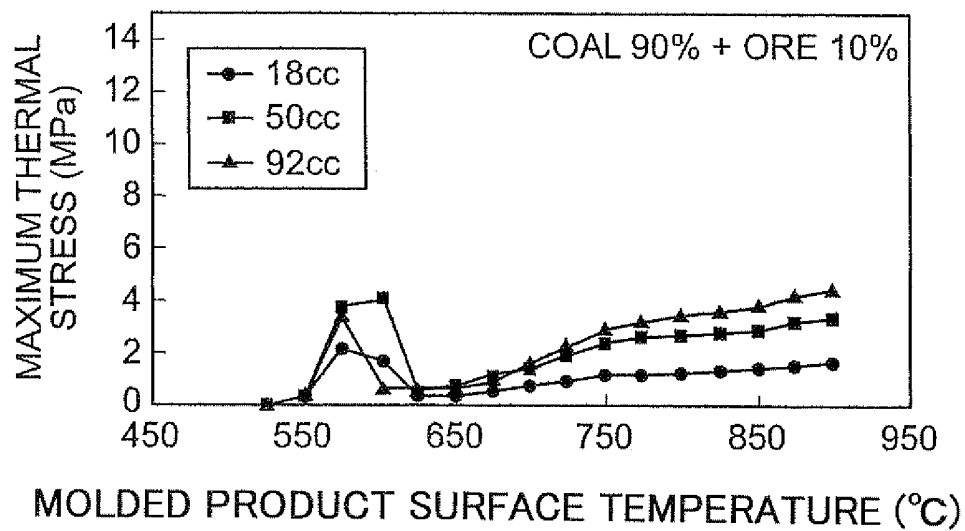


FIG.3

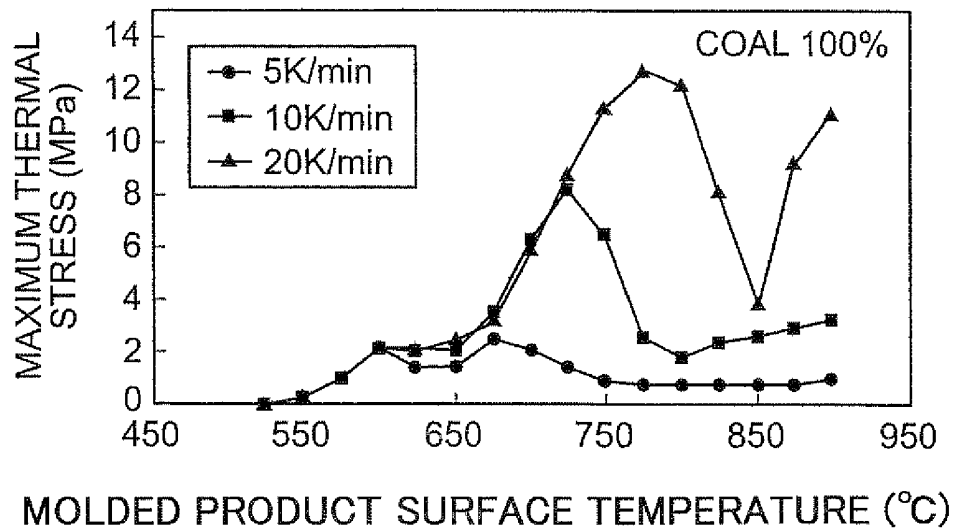


FIG.4

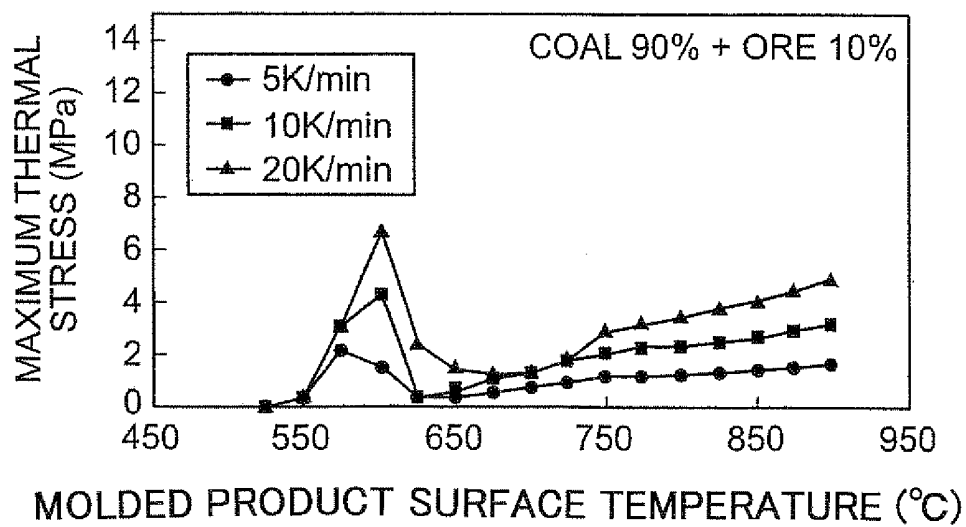
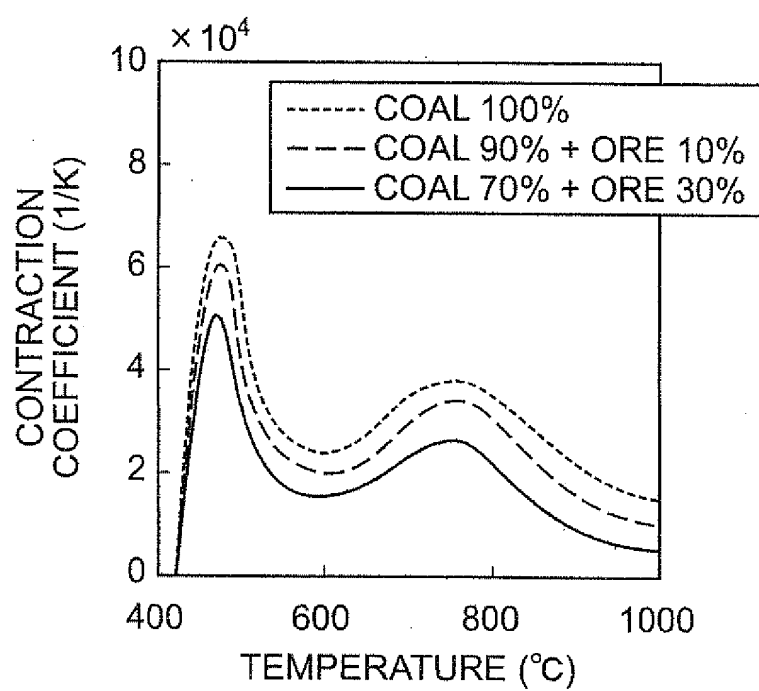


FIG.5



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2007/075392

A. CLASSIFICATION OF SUBJECT MATTER

C10B57/06(2006.01) i, C10B53/08(2006.01) i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C10B1/00-57/18

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho	1922-1996	Jitsuyo Shinan Toroku Koho	1996-2008
Kokai Jitsuyo Shinan Koho	1971-2008	Toroku Jitsuyo Shinan Koho	1994-2008

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 6-65579 A (Nippon Steel Corp.), 08 March, 1994 (08.03.94), Claims; Par. Nos. [0017] to [0028]; table 2 (Family: none)	1
Y	JP 56-47234 B2 (Nippon Steel Corp.), 09 November, 1981 (09.11.81), Fig. 1; page 1, column 1, line 33 to column 2, line 17 & JP 52-23102 A	1
Y	JP 54-148001 A (Sumikin Kako Kabushiki Kaisha), 19 November, 1979 (19.11.79), Claims; page 3, upper left column, lines 8 to 14 (Family: none)	1

☒ Further documents are listed in the continuation of Box C.
 ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

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"&" document member of the same patent family

Date of the actual completion of the international search
31 March, 2008 (31.03.08)Date of mailing of the international search report
08 April, 2008 (08.04.08)Name and mailing address of the ISA/
Japanese Patent Office

Authorized officer

Facsimile No.

Telephone No.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2007/075392

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
E, X	JP 2008-13637 A (JFE Steel Corp.), 24 January, 2008 (24.01.08), Scope of Claims; Claim 1 (Family: none)	1

Form PCT/ISA/210 (continuation of second sheet) (April 2007)

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2007/075392

Claim 1 involves any method so long as the carbonization is carried out while controlling the heating rate to $20^{\circ}\text{C}/\text{min}$ or less in a temperature zone where the surface temperature of the molded article is from 550 to 650°C , regardless of the heating rate in a temperature range other than 550 to 650°C .

In contrast, the description presents no statement on the heating rate in a temperature range other than 550 to 650°C and "the heating rate in a temperature range other than 550 to 650°C is appropriately changed and no constant heating rate is employed at the individual temperatures" appears in Example 1. That is to say, the heating rates in the individual temperature ranges of the molded article surface temperature vary from range to range in the cases which are described in detail in the description and in which the shape retention ratios are evaluated. In these cases, therefore, the heating rate conditions in temperature ranges other than 550 to 650°C are unclear.

In producing molded cokes, on the other hand, it is a publicly known technique that there is a desirable heating rate range depending on heating temperature (see JP 56-47234 B2 (Nippon Steel Corp.), Fig. 1, page 1, column 1, line 33 to column 2, line 17 cited in Box C)

Thus, neither the statement in the description nor the statement in Examples seems reasonable as a ground for the coke can be prevented deformation or cracking merely by considering the heating rate in the temperature range of 550 to 650°C as the specific condition regardless of the heating rates in other temperature ranges.

Such being the case, claim 1 is not fully supported in the meaning within PCT Article 6.

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

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