

SHINAGAWA TECHNICAL REPORT

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品川技報

報文 REPORT

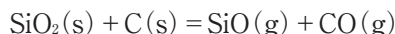
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Influences of SiO₂ Component and Preheating History on Corrosion Rate of Slag Line Castable for Blast Furnace Main Trough

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要 旨

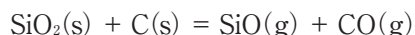
高炉主樋SL用SiC-Al₂O₃-C質の流し込み材のSiO₂含有量と事前加熱が損耗速度に及ぼす影響を侵食試験で評価した。その結果、SiO₂含有量に比例して損耗速度は大きくなり、その傾向は事前加熱することで顕著になった。一方、化学分析の結果、事前加熱により材料中のCが減少すること、Cの減少はSiO₂含有量に比例して顕著になることが示された。これはSiO₂がCを酸化する以下の反応によるものと考えられた。



これより、事前加熱がSiO₂の損耗促進を助長したのは耐食性向上効果の高いCを酸化して減少させたためと推定された。実樋において主樋耐火物は背面側で高温の事前加熱を長時間受けた後に稼働面として露出することから、背面側での事前加熱中にSiO₂によるCの酸化が生じ、耐食性の低下が誘発されている可能性が考えられた。これより、損耗速度を低減させるには材料中のSiO₂を可能な限り減らすことが重要であることがわかった。

Abstract

A corrosion test for evaluating the influences of SiO₂ content and preheating history on the corrosion rate of SiC-Al₂O₃-C castable refractory for main trough in blast furnace was carried out. It was clarified that, in accordance with an increase of SiO₂ content, the corrosion rate of the castable refractory specimen was accelerated with a tendency, preheating for such reduction in the corrosion resistance to become more prominent. It was also understood through chemical analysis that the C content in a specimen is lowered by firing with marked dependency on the coexisting SiO₂ content. The reduction of the C content caused by firing can be attributed to oxidization of C by SiO₂ component, which is expressed with the following chemical equation.



By combining the above two experiment results, it is inferred that the accelerated corrosion

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of castable refractory specimens with a preheating history was caused by the reduction of the C component of which the existence is effective for securing corrosion resistance. Taking into account the time-series wearing situation of the main trough castable refractories in an actual blast furnace operation that, after gradual wear-off of the hot face refractories over a relatively lengthy time period, the back portion of refractory is exposed to molten metal and slag as the hot face. It can be predicted that, due to oxidation of the C component by SiO_2 until surfacing at the hot face, the corrosion resistance of newly surfaced refractory has been reduced to some extent. It was concluded that minimizing the SiO_2 content in castable refractories is important to improve or stabilize the corrosion rate.

1. 緒言

高炉から排出された溶銑と溶融スラグは、主樋で比重分離され、溶銑樋、スラグ樋を通して次工程に移される。主樋は溶銑滓を最初に受ける樋であり、多くは「貯銑式」と呼ばれる溶融物を常に貯めておくような構造をしている。主樋は、高炉の樋の中でサイズが最も大きく耐火物の損傷が最も激しいため、高炉の耐火物原単位を低減するためには主樋の寿命延長が重要である。

主樋の寿命を律速するのはメタル-スラグ界面（以下、ML）とスラグ-大気界面（以下、SL）の2つの局部損耗部位で、これらの損耗は化学的溶損で生じている^{1,2)}。そのため、ML、SLそれぞれの損耗メカニズムに対応する材質を適用したゾンドライニング施工が国内の主樋で一般的に実施されている（Fig.1）。その内、SL部に適用されるSL材は、溶銑と接触しないことから、スラグに対して高耐食性を示すSiCを多量に含むSiC- Al_2O_3 -カーボン（以下、C）材質が使用される。

1. Introduction

Molten pig iron and molten slag tapped from the blast furnace are separated by utilizing the difference in their specific gravities in the main trough. This led to hot metal runner and slag runner, respectively. The blast furnace main trough into which molten pig iron and slag are simultaneously tapped is generally designed to be “hot metal storage” type so as to hold hot metal. Since the main trough is characterized by its maximum trough size and severely damaged portion of relined refractories, securing its refractory service life is essential for minimizing blast furnace refractory consumption.

The service life of main trough refractories is regulated by localized wear at the metal-slag interface (hereinafter referred to as ML) and at the slag-atmosphere interface (hereinafter referred to as SL) induced by chemical corrosion^{1,2)}. Due to this reason, as shown in Fig. 1, zoned lining method in which two kinds of refractories, each of which is designated to be

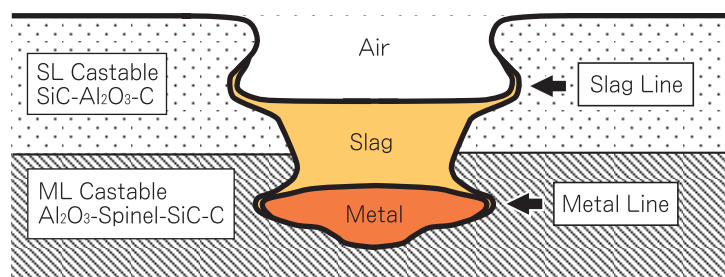


Fig. 1 Zoned lining of blast furnace main trough with ML and SL castable refractory.

SiCに加えてCも耐食性の向上に有効で、酸化等によりC含有量が低下すると耐食性は著しく悪化する^{3,4)}。その他の成分として、キャストブルの流動性改善、組織の緻密化、焼結促進等を目的にシリカフューム（以下SF）等のシリカ超微粉が添加されることもある。しかし、シリカ原料の添加により耐食性は低下することが一般的に知られている。さらに、SiO₂成分がCを酸化することが報告されている⁵⁾が、耐食性への影響について評価されたことはなかった。

出銑および貯銑中、主樋の稼働面は高温の溶融物に曝され続け、稼働面にごく近い背面部も高温で加熱されている。損耗速度を考慮すると、稼働面は高温で長時間事前加熱された後に露出していることになる。溶銑滓の平均出銑温度は1500℃から1530℃程度であるが、個々の出銑においては1600℃に近い温度になる場合がある。そのため、さまざまな熱履歴を受けた後に稼働面として露出する状態を考慮して材質を評価することが必要であり、主樋ML材では事前加熱による材料特性の変化が損耗速度に与える影響について報告されている⁶⁾。しかし、SL材について評価した例は報告されていない。

これらより、配合中のSiO₂成分量、事前加熱、および反応温度が主樋SL材の損耗速度に与える影響について調査した。

durable to a corresponding corrosion mechanism, are generally applied in the blast furnace trough. At the SL region where relined refractories do not directly contact with hot metal, SiC-Al₂O₃-C castable refractories containing a large amount of SiC, which are characterized by superior slag corrosion resistance, are applied.

Similar to SiC, C component is effective for securing and improving corrosion resistance. When the C content in castable refractories is lowered because of oxidation, corrosion resistance is accordingly reduced^{3,4)}. For the purposes of securing fluidity of castable refractories during relining work, densification of the relined structure and/or promotion of sintering reaction, fine silica particles, such as silica fume (hereinafter referred to as SF) are added to castable refractories. It is generally acknowledged, however, that corrosion resistance of castable refractories is deteriorated by silica addition. In addition, oxidation of C component by SiO₂ was reported⁵⁾, however, its influence on corrosion resistance has not been evaluated in detail.

In actual operation at the blast furnace, the hot face of main trough refractory is always exposed to molten metal/slag at high temperatures during hot metal tapping and holding. This influences the back end of the relined refractories with providing thermal energy for heating up. Trough refractory wear progresses, the newly surfaced hot face is exposed to hot temperature conditions. This

Table 1 Compositions of samples used for investigation in mass%

Raw materials	Grain size	A	B	C	D
Silicon carbide	+1 mm	47	47	47	47
Silicon carbide	1 mm-75 μ m	22	22	22	22
Silicon carbide	-75 μ m	16	16	16	16
Calcined alumina		9	8	6	4
Silica fume		0	1	3	5
Carbon raw materials		4	4	4	4
Alumina cement		2	2	2	2
Additives		0.17	0.17	0.17	0.17
Mixing water		5.2	5.2	5.2	5.2

2. 試験内容

2.1 供試試料

Table 1に試料の配合組成を示す。試料Aは、粗粒、細粒にSiCを、微粉にはSiC、仮焼アルミナ、C原料を使用し、バインダーとしてアルミナセメントを使用した低セメントキャストブルである。試料B, C, Dは、試料Aから仮焼アルミナを減量し、SFを1, 3, 5mass%置換することにより配合組成中のSiO₂成分量を変化させた。なお、基礎的な実験であるため通常添加される酸化防止剤、爆裂防止剤等は無添加とした。

各試料は、万能ミキサーで3分間混練した後所定の枠に casting 込み、室温で24h養生後、110℃で24h乾燥を行った。混練水量については、耐食性への水分添加量の影響を排除するため、各配合共5.2mass%一定とした。乾燥後の試料は、コークスブリーズ中に詰め、電気炉を用いて、所定の温度、時間の加熱を行った。

2.2 試料の分析

試料A～Dの加熱後試料を微粉碎し、C成分の定量分析を行った。

2.3 侵食試験

耐食性評価はアーク加熱回転ドラム侵食試験法により行った。試験方法の模式図をFig.2に示す。侵食剤には1.2kgの高炉スラグを使用し、1時間毎に侵食剤を交換した。試験前後の試料厚みを測定することで侵食量を算出し、損耗速度で評価した。

results in unintentional lengthy preheating time. Average hot metal tapping temperature ranged from 1500 °C to 1530 °C with sporadic exceptions of roughly 1600 °C. Therefore, it is important to evaluate the characteristics of castable refractories with prospect that the newly surfaced hot face of trough castable refractories has experienced various preheating conditions. Investigations on the influence of altered castable refractory characteristics induced by preheating on the corrosion rate of ML castable refractories have been reported⁶⁾. However, similar investigations on SL castable refractories have not been conducted. The influences of SiO₂ content, preheating history and reaction temperature on the corrosion rate of SL castable refractories are evaluated in this investigation work.

2. Experiments

2.1 Preparation of samples

The compositions of 4 types of SL castable refractory samples are summarized in Table 1. Sample A is low cement castable which is composed of mainly coarse and fine SiC particles combined with fine powdery raw material of SiC, calcined alumina, C and, as binder, alumina cement. In samples B, C and D, 1, 3 and 5 mass% SF was substituted for part of the calcined alumina, respectively. Because the prepared samples were used only for experiments in the laboratory, neither an antioxidant nor explosion

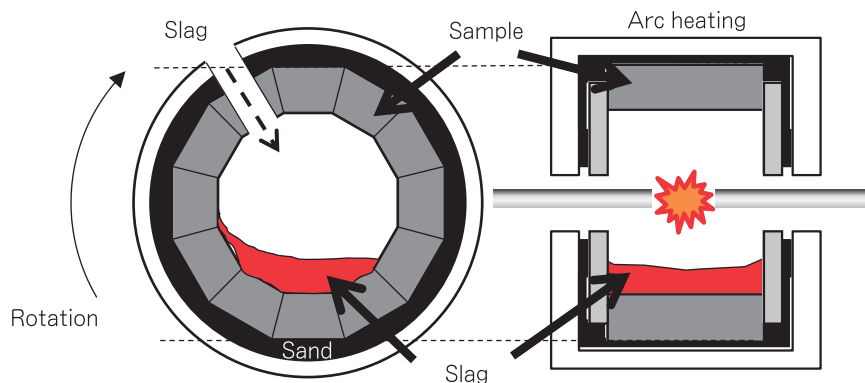


Fig. 2 Schematic drawing of rotary drum corrosion test.

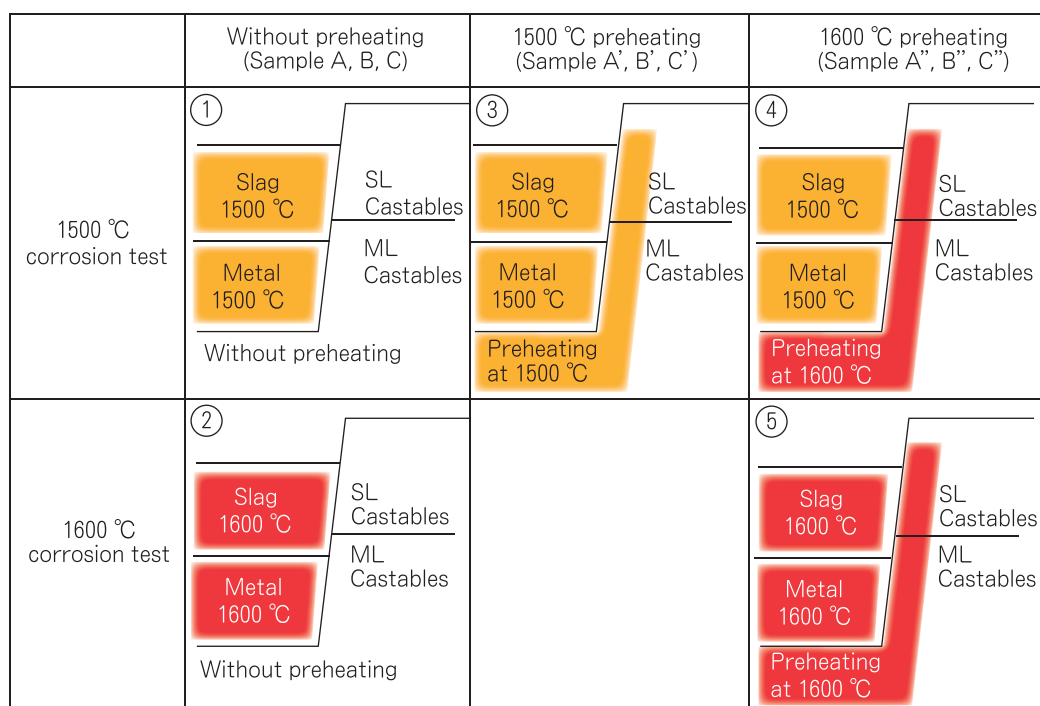


Fig. 3 Simulative conditions for series of corrosion tests.

試験はFig.3に示すように、①～⑤の5つの場合を想定し実施した。1500℃は通常の出鉄温度の操業を、1600℃は高い出鉄温度の操業を想定している。事前加熱は稼働面近傍の背面側で出鉄による熱履歴を受けた後に稼働面として露出する状態を想定している。①、②では、侵食試験温度とSiO₂成分が熱履歴を受ける前の樋材の損耗速度に与える影響を評価した。③、④、⑤では、侵食試験温度とSiO₂成分が熱履歴を受けた後の樋材の損耗速度に与える影響を評価した。③は、通常温度の事前加熱後に通常温度の出鉄を受ける場合を、④は高温の事前加熱後に通常温度の出鉄を受ける場合、⑤は高温の事前加熱後に高温出鉄を受ける場合を想定している。

3. 試験結果

3.1 化学分析

Fig.4に加熱後試料のC成分の化学分析結果を示す。SF添加量に比例して加熱後のC含有量が低下した。その傾向は1600℃加熱後試料でより顕著であった。

preventing agent was added.

Each sample was mixed with a universal mixer for 3 minutes, followed by casting into a designated mold. After curing at room temperature for 24 hours, the demolded samples were dried at 110 °C for 24 hours. It should be noted that every sample was mixed with 5.2 mass% of constant water content so as to avoid any influences of added amount of water on the corrosion rate of the samples. After drying, the sample specimens packed in a coke breeze were fired at 1500 °C or 1600 °C for a specified time period in an electric furnace.

2.2 Analysis of C content in fired specimens

The C content is quantitatively analyzed, using crushed specimen of each fired sample.

2.3 Corrosion test

Corrosion resistance was evaluated with a rotary drum corrosion test with arc heating for 5 hours, which is schematically illustrated in Fig. 2. 1.2 kg of blast furnace slag was added as the corrosion agent and it was exchanged

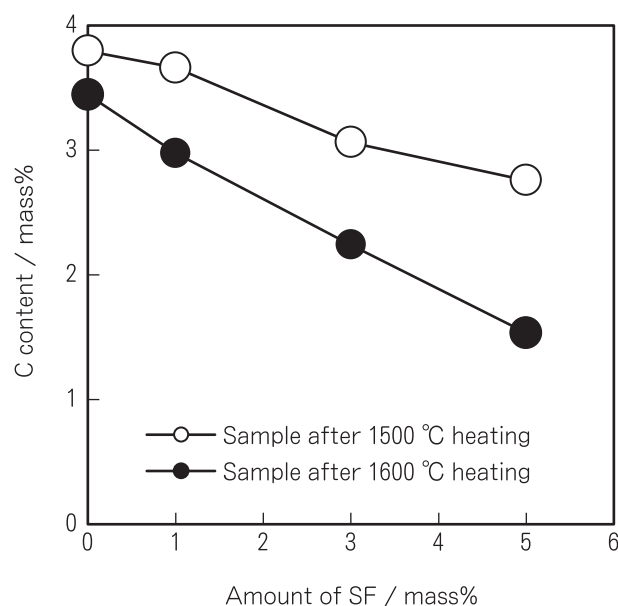


Fig. 4 Influence of initial SF content and firing temperature on C content after heating.

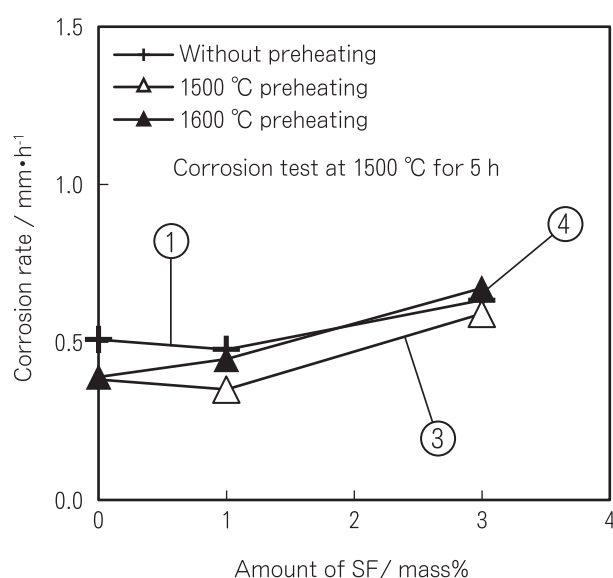


Fig. 5 Change of corrosion rate as a function of initial SF content in corrosion test under simulative condition ①, ③ and ④.

3. 2 侵食試験

Fig. 5に1500℃で侵食試験を行った条件①, ③, ④の結果を示す。1500℃の試験では事前加熱の有無や事前加熱の温度によらず同程度の損耗速度を示した。また, SF添加量に比例して損耗速度は幾分増大した。

every one hour. The corrosion resistance of the sample was evaluated with corrosion rate which was calculated by the remaining thickness of the specimen after the corrosion test.

A series of corrosion tests were conducted with the five (5) simulative conditions illustrated in Fig. 3. Hot metal tapping operation with normal and high tapping temperatures was simulated with 1500 °C and 1600 °C of corrosion test temperature, respectively. Preheating history at normal and high temperature condition for the newly surfaced trough refractory was simulated with 1500 °C and 1600 °C of firing temperature for the sample specimens. In the corrosion test under simulative condition ① and ②, the influences of corrosion test temperature and SiO₂ content on the corrosion rate of the sample with no preheating history were evaluated. In the corrosion test under simulative condition ③, ④ and ⑤, the influences of corrosion test temperature and SiO₂ content on corrosion rate of samples which had a preheating history at 1500 °C or 1600 °C were evaluated. Simulative conditions ③, ④ and ⑤ indicate normal tapping temperature operation with trough refractories having a preheating history at normal temperature, normal tapping temperature operation of trough refractories with preheating history at high temperature and high tapping temperature operation of trough refractory with preheating history at high temperature, respectively.

3. Results of Experiments

3. 1 Analysis of C content in fired specimens

Fig. 4 shows the relation between initial SF content and C content in samples which were fired at 1500 °C or 1600 °C. In accordance with the increase of initial SF content, the C content was lowered. Sample specimens fired at 1600 °C exhibits more marked tendency of C content reduction.

3. 2 Corrosion test

Fig. 5 shows the corrosion test results under ①, ③ and ④ simulative conditions, all conducted at 1500 °C corrosion test temperature.

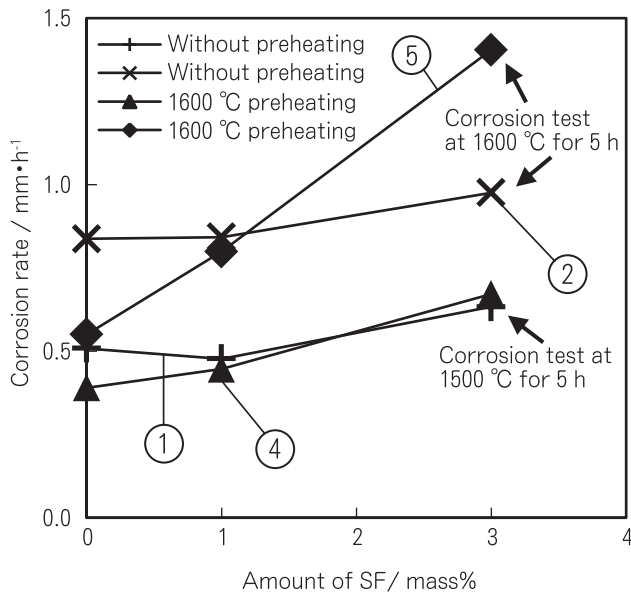


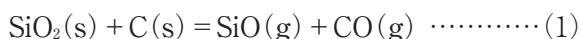
Fig. 6 Change of corrosion rate as a function of initial SF content in corrosion test under simulative condition ①, ②, ④ and ⑤.

Fig. 6に、Fig.5の条件①、④の結果に1600℃で侵食試験を行った条件②、⑤の結果を加えた図を示す。条件①、②の比較より、事前加熱していない試料では1600℃における侵食試験の方が1500℃における試験より大きい損耗速度を示すこと、SF添加量に対する損耗速度の傾きは試験温度によらず同程度であることがわかる。一方、1600℃加熱後試料を用いた条件④、⑤の結果を比較すると、1600℃における侵食試験の方が1500℃における試験より大きい損耗速度を示し、かつ、損耗速度に与えるSF添加量の影響も顕著となることがわかる。

4. 考察

4. 1 化学分析

Fig.4で、加熱後のCはSF含有量に比例して減少することから、Cの減少は式(1)に示すSiO₂の還元反応、すなわちSiO₂によるCの酸化、によるものと考えられる。



この反応は大気圧下で反応に参与する元素がSi、C、Oのみの場合、1525℃以上で右に進行する

Regardless of preheating history or temperature in preheating history, all the sample specimens exhibited almost equivalent corrosion rates with a tendency that, in accordance with the increase of initial SF content, the corrosion rate slightly worsened.

The corrosion test results under simulative conditions ② and ⑤ (at 1600 °C of corrosion test temperature) are shown in Fig. 6, overlapping the corrosion test results under simulative conditions ① and ④ (at 1500 °C of corrosion test temperature). Comparison of corrosion test results under simulative conditions ① and ②, in which the sample specimens with no preheating history were evaluated, indicates that the corrosion rate under corrosion test temperature 1600 °C was bigger than that under 1500 °C of corrosion test temperature with similar dependency on corrosion rate incrementation of initial SF content. Comparison of corrosion test results under simulative conditions ④ and ⑤, in which the samples with a preheating history at 1600 °C were evaluated, indicates that the corrosion rate under 1600 °C of corrosion test temperature was much bigger than that under 1500 °C of corrosion test temperature with more prominent influence of initial SF content on corrosion rate incrementation.

4. Discussion

4. 1 Analysis of C content in fired specimens

The conclusion derived from Fig. 4 is that, in accordance with an increase of initial SF content, the C content in the sample specimen is lower after firing. This can be explained by the oxidation of C component by SiO₂. The chemical equation is expressed with equation (1).



It is known that, under the circumstance in which the three elements Si, C and O solely take part in the oxidation of carbon under ambient pressure, the reaction expressed with equation (1) makes progress toward the right-hand side over 1520 °C⁵⁾. Reduction of C content

ことが知られている⁵⁾。今回の実験で1500℃加熱でもCが減少したことについて、SL材中にはCa等の成分も含まれており多くの元素が関与しているため、SiO₂によるCの酸化が起こる温度がいくぶん低下しているとするのが妥当であろう。一般的に、反応速度は温度が高い程速いため1600℃加熱後の方がSF添加量の影響が強くなったと推定される。加熱時のコークスブリーズ中は大気圧であることから、式(1)で生成したCOガスは系外に散逸すると考えられる。

4. 2 侵食試験

Fig.6で、事前加熱によらず1600℃での試験が1500℃での試験に比較して大きい損耗速度を示したのは温度が高く総括反応速度が大きくなったためで妥当な結果である^{7,8)}。事前加熱によりSFの損耗促進効果が助長されたのは、SiO₂の耐食性低下効果よりも事前加熱時のSiO₂によるCの酸化に伴う耐食性低下の影響の方が強いと考えられる。

安尾らはSiCを76%含む低セメントキャストブルを用いて、C原料添加量と損耗速度の関係を示している(Fig.7)³⁾。報告によると、C原料添加量が低下する程、1500℃と1600℃侵食試験の損耗速度

by firing even at 1500℃ can be explained with inference that, since various elements contained in SL castable refractories, such as Ca, take part in the reaction, C oxidation temperature is lowered to some extent. It is inferred that, because of a general tendency for the reaction to become more active in proportional to the reaction temperature, the influence of the initial SF content on reduction of C content becomes more marked. It is safe to conclude that, since the sample specimens packed in a coke breeze were fired under ambient pressure, CO gas generated by equation (1) were volatilized out of the system.

4. 2 Corrosion test

The experimented result was that, as is easily concluded with Fig. 6, the corrosion rate in the corrosion test at 1600℃ is bigger than that observed in corrosion test at 1500℃^{7,8)}. This can be explained by more active reactivity, or a higher overall reaction rate caused by temperature rise. Influence of the initial SF content on corrosion rate of the fired sample specimens can be attributed to reduction of their corrosion resistance caused by oxidation of C by SiO₂ rather than negative influence of the contained SiO₂ on corrosion resistance.

As per Fig. 7, Yasuo et al. reported the relationship between the C content in 76% SiC containing low cement castable and the corrosion rate³⁾. According to their report, in proportion to lowering the C content, the difference in the corrosion rate between 1500℃ and 1600℃ of corrosion test temperature becomes bigger. The corrosion rate of samples simply dried at 110℃ in corrosion test under simulative conditions ① and ② as well as the corrosion rate of samples fired at 1600℃ under simulative conditions ④ and ⑤ are plotted in Fig. 8 as a function of C content of the samples. The test results under simulative conditions ④ and ⑤, in which the C content analyzed on fired specimen is used, are similar to the relationship reported by Yasuo et al. The corrosion rates of the not fired samples in the corrosion test under simulative conditions ① and ② are plotted at 4% of the initial

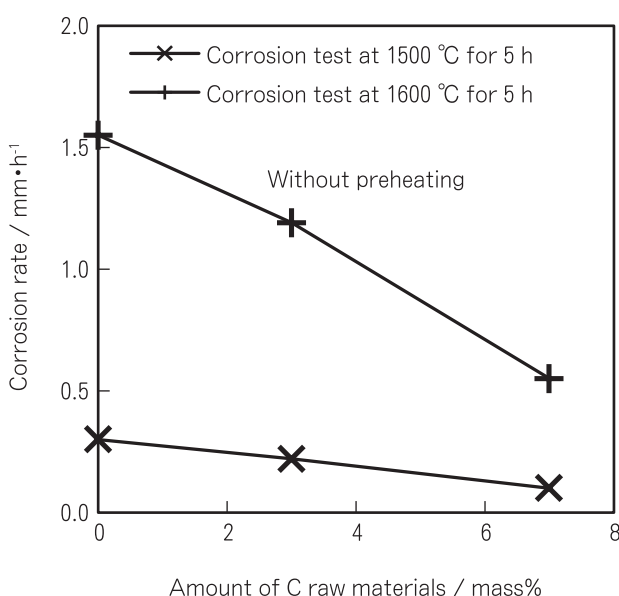


Fig. 7 Influence of raw C material content on corrosion rate³⁾.

の差は大きくなる。Fig.8に、条件①、②の110℃加熱後試料と条件④、⑤の1600℃加熱後試料の損耗速度をC含有量に対してプロットして示す。条件④、⑤の試験結果には、安尾らの報告と同様の傾向が確認された。Fig.8には事前加熱していない試料の損耗速度をC量 4 mass%の位置にプロットした(条件①、②)。これからも、 SiO_2 量よりC量の影響が大きいことがわかる。

これらに対し、1500℃で試験したFig.5で事前加熱が損耗速度に影響しなかったのは、試験温度が低く総括反応速度が小さいためそれぞれの試料の耐食性の差が顕在化しなかったためであろう。

高炉操業において出銑温度は変動し、高温出銑時に損耗速度が大きくなることは容易に推定できる。今回の実験より、主樋材の溶損を検討する際は溶損時の反応速度に加えて事前加熱による稼働面の耐食性変化を考慮する必要があることがわかった。また、SL材を設計する際、耐食性向上効果のあるCを添加するとともに、事前加熱時にCを酸化し材料の耐食性を低下させる SiO_2 の添加量は可能な限り少なくすることが好ましいと考えられる。

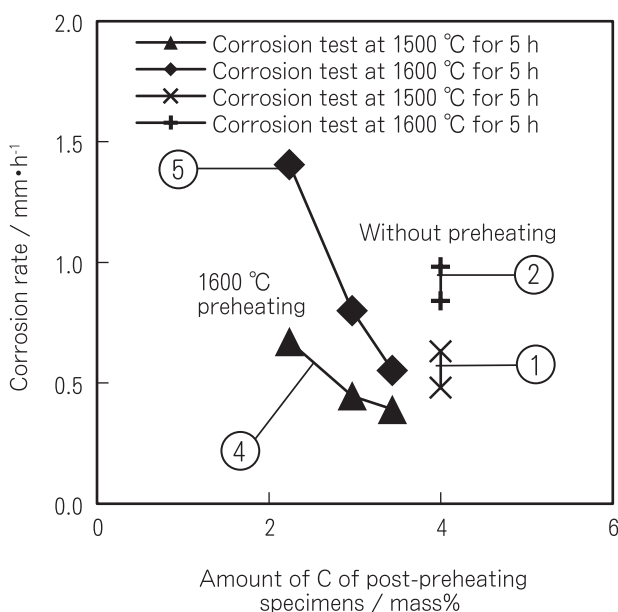


Fig. 8 Changes of corrosion rate as a function of C content in corrosion test under simulative condition ①, ②, ④ and ⑤.

C content in Fig. 8. It is obvious in Fig. 8, that the negative influence of C content on corrosion resistance is more prominent than that of SiO_2 content.

As shown in Fig. 5, marked influence of preheating history on the corrosion rate is not observable in corrosion test at 1500 °C. It is inferred that, because of small overall reaction rate at low corrosion test temperatures, the difference in corrosion resistance between the samples is not tangibly actualized.

With the possibility that main the trough refractories could be damaged more severely during high temperature hot metal tapping with the nature of fluctuating tapping temperature in blast furnace operation, it became clear in this investigation that the corrosion behavior of the main trough refractory needs to be evaluated considering not only the corrosion reaction rate but also the deteriorated corrosion resistance caused by preheating history. It was also clarified that it is necessary for design work on SL castable refractory to add appropriate amount of C component so as to ensure corrosion resistance as well as to minimize the added amount of SiO_2 which, during unavoidable preheating period in campaign life, could deteriorate corrosion resistance by oxidizing the C component.

5. Conclusions

Through a rotary drum corrosion test on $\text{SiC-Al}_2\text{O}_3\text{-C}$ castable refractory sample specimens for investigating the influences of the SiO_2 content, preheating history and reaction temperature on the corrosion rate of SL castable refractories applied to the blast furnace main trough, the following conclusions were obtained.

- 1) By firing SL castable refractory sample specimens in a coke breeze, the C content was lowered in proportion to the initial SF content with a prominent tendency that, in accordance with the rise in firing temperature, the reduction of C content becomes bigger. The reduction in C content can be attributed to the oxidation of the C

5. 結言

高炉主樋SL材の損耗速度に及ぼす SiO_2 含有量、事前加熱、および反応温度の影響をアーク加熱回転ドラム侵食試験で調査し、以下の知見を得た。

- 1) SL材をコークスブリーズ中で加熱した場合、SF添加量に比例してCが減少し、その傾向は高温ほど顕著となった。これは SiO_2 がCを酸化しているためと考えられた。
 - 2) 1600℃での試験の結果、材料中のSF量が増えるほど損耗速度が大きくなり、その影響は事前加熱試料で大きくなった。これは事前加熱時に SiO_2 がCを酸化、C減量により材料の耐食性が低下したためと推定された。
 - 3) 1500℃での試験では事前加熱が損耗速度に大きな影響を与えなかった。温度が低く総括反応速度が小さいため材料の耐食性の差が顕在化しなかったと考えられる。
 - 4) 実炉においてSL材の損耗を考える場合、反応温度のみでなく事前加熱温度による稼働面の耐食性変化も考慮する必要がある。
 - 5) 高温出銑に対応する主樋SL材を開発する場合、Cを添加するとともに、 SiO_2 成分を可能な限り減らすことが重要である。
- component by SiO_2 .
- 2) In the corrosion test at 1600℃, the corrosion rate increased in proportion to the initial SF content. Especially, sample specimens with preheating history exhibited a bigger corrosion rate. It is inferred that the higher corrosion rate of sample specimen with preheating history is caused by the deterioration of corrosion resistance induced by the lowering of C content resulting from oxidation by SiO_2 .
 - 3) In the corrosion test at 1500℃, marked influence of preheating history on corrosion rate was not observable. It is inferred that, because of the small overall reaction rate at low corrosion test temperature, the difference in corrosion resistance between the sample specimens was not tangibly actualized.
 - 4) When studying corrosion behaviors of trough refractories in actual blast furnace operation, it is necessary to consider not only the reaction temperature but also the altered corrosion resistance of the newly surfaced refractories which might have been deteriorated by preheating history.
 - 5) In order to develop SL castable refractories with sufficient durability for high tapping temperature operation, it is essential to add the appropriated amount of C component as well as to minimize the SiO_2 content.

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