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Microwave Drying of Monolithic Refractories

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Abstract

Monolithic refractory technology has been developed for ladles and RH vessels, with the purpose of reducing the total refractory cost in Nippon Steel Corp. The technology includes mixing, installing by casting, curing, drying. One of the most important things is drying shortly without explosion. In concerning with this drying, we developed the combination drying method using microwaves and hot air. Heating by microwave is internal heating, therefore, it is easier rapid heating until back of structure with appropriate microwave power applied. This paper reports on drying behavior of Al_2O_3-MgO, and Al_2O_3-Spinel monolithic refractories used with ladle and RH vessels and introductory situation of the microwave equipment in Nippon Steel Corp.

1. Introduction

The refractories that are used for steelmaking equipment are divided into monolithic refractories and bricks. Today, at each of Nippon Steel's steelworks, the proportion of monolithic refractories has increased to as much as 70% to 80% from around just 30% in the 1960s. This is due largely to the saving of energy made possible by the use of monolithic refractory and the saving of labor made possible by mechanization of refractory work. The application of monolithic refractory to ladles is a typical example.

Specifically, the cast-in refractory method using a core was developed in 1978.[1] The salient feature of this method is that it permits mixing, pressure feeding, forming, curing and drying of the refractory in a systematic manner. In effect, it is a new technology that mechanizes and systematizes the operational elements involved in the conventional monolithic refractory casting-in process. This method uses a vortex flow-type mixer for mixing, a hydraulic diaphragm pump for pressure feeding, and a rod vibrator to remove air.

Umeya's paper can be cited as an authentic technical commentary on the theory of monolithic refractory work.[2] Measurement of the rheologic characteristics of monolithic refractory discussed in that paper has contributed much to the improvement in monolithic refractory work.

Another important element in monolithic refractory work is the technology for drying monolithic refractories. Namely, in conventional bricklaying, it is only necessary to dry the joint mortar in a comparatively short time and then preheat the bricks. By contrast, since monolithic refractory must be wetted with water while being worked, it is indispensable to take time to dry it out once the work is complete.

To dry the monolithic refractory, a hot air burner, which has long been used to dry and preheat conventional brick ladles, is employed. However, since the considerable length of time required to dry the monolithic refractory in this manner was considered problematic, extensive R&D on the drying of monolithic refractory has been carried out. Attempts to dry monolithic refractory too quickly can cause the refractory to explode—a common problem with refractory materials. With the aim of clarifying the causes of this unwanted phenomenon, various approaches have been taken, such as estimating the vapor pressure in the refractory during the drying process,[3] using a drying/heat transfer model,[4] and studying the explosive characteristics.[5] In addition, the relationship between the internal vapor pressure and material strength of the refractory, which was used for a quantitative evaluation of the phenomenon,[6] has also been reported.

As an alternative means of drying monolithic refractory more efficiently, the hot air & microwave drying process has been reported.[7] According to the report, the microwave frequency used to dry monolithic refractory was 915 MHz, and the monolithic refractory tested

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was a zircon-based type. Reportedly, this drying process reduced the drying time to about one-third and cut energy consumption to about one-fourth. Incidentally, the report focuses on the practical application of the process and only briefly discusses the interactions of microwave and monolithic refractory.

Therefore, we experimentally applied the hot air & microwave drying process to monolithic refractories that are currently used for ladles and RH vessels (e.g. alumina-magnesia and alumina-spinel refractories). The frequency of the microwave used was 2.45 GHz. This paper describes those experimental results. It also describes the current status concerning introduction of the microwave drying equipment at Nippon Steel.

2. Microwaves

2.1 Definition of microwave

The definition of microwave is shown schematically in **Fig. 1**. Thus, microwave refers collectively to electromagnetic waves in the frequency range 300 MHz to 300 GHz. Nippon Steel has adopted 915 MHz and 2.45 GHz as the microwave frequency bands. In the present experiment, we used a microwave of 2.45 GHz, the same frequency as used in microwave ovens.

2.2 Principle and characteristics of microwave heating

2.2.1 Principle of microwave heating

The principle of microwave heating is shown in **Fig. 2**. When microwaves irradiate the monolithic refractory lining on the inside of a ladle or RH vessel, the dipoles of the raw material of the monolithic refractory and the moisture contained within vibrate and rotate, producing an internal friction. The resulting frictional heat raises the temperature of the monolithic refractory. After the moisture has evaporated completely, all the energy of the irradiating microwave is absorbed by the refractory raw material. As a result, the refractory temperature continues rising further.

2.2.2 Characteristics of microwave heating

Assuming the microwave energy that is transformed into heat inside a certain material as P (W/m^3), P can be expressed as follows.

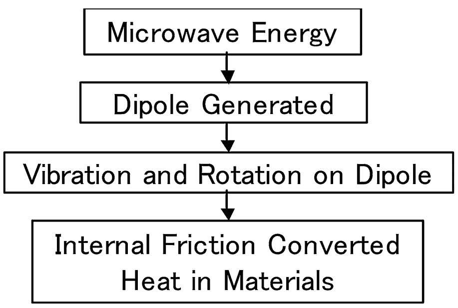

Fig. 2 Principle of microwave heating

$$P \propto f * E^2 * \varepsilon\gamma * \tan\delta$$

In the above expression, the terms, f: frequency (Hz) and E: electric field intensity (V/m), represent the equipment factors, and the terms, $\varepsilon\gamma$: specific dielectric constant and $\tan\delta$: angle of dielectric loss, represent the material factors. The material factors are dependent not only on the material but also on the frequency and temperature. The salient characteristics of microwave heating are: (1) internal heating, (2) selective heating, and (3) controlled heat-up rate.

3. Experimental Procedure

3.1 Microwave drying equipment

Fig. 3 shows the system configuration for microwave drying. The applicator used measures 1,500 mm in width, 1,500 mm in length and 2,500 mm in height, all being inner dimensions. All other system components are readily available on the market. The frequency and maximum output of the microwave generator are 2.45 GHz and 5 kW, respectively. The atmospheric temperature inside the applicator can be set to a prescribed value by the hot air generator (electric heater).

3.2 Preparation of samples for drying test

Alumina-magnesia monolithic refractory is commonly used for ladle sidewalls and tuyeres, etc. The chemical composition of the alumina-magnesia monolithic refractor tested is shown in **Table 1**. As raw materials for the alumina, an aggregate containing more than 99.5 mass% Al_2O_3 and calcined alumina were used. As the raw material for magnesia, an aggregate containing more than 97.3 mass% MgO was adopted.

Alumina-spinel monolithic refractory is commonly used for the bottoms of ladles and the lower vessels of RH, etc. The chemical composition of the alumina-spinel monolithic refractory that was subjected to a drying test is shown in **Table 2**. As raw materials for the alumina, an aggregate containing more than 99.5 mass% Al_2O_3 and calcined alumina was used. As the raw material for spinel, an aggregate containing more than 99.3 mass% Al_2O_3 and MgO was used.

The test samples were prepared by the following method. Concerning the alumina-magnesia monolithic refractory, the raw materials were first mixed with 5.6-6.3 mass% water using a small, universal mixer and then poured into a metallic form measuring 200 mm × 200 mm × 200 mm. After the mixture was formed on a vibrating table, it was cured for 24 hours. The refractory temperature was measured using K-thermocouples installed on the surface, in the center (100 mm from each side) and at the bottom of each sample. The internal pressure of the refractory was measured during drying by a copper tube (2 mm in OD, 1 mm in ID), which was embedded near the thermocouple in the center of the sample when the sample was formed.

As for the alumina-spinel monolithic refractory, the raw materials were first mixed with 4.3 mass% water (a fixed amount of water

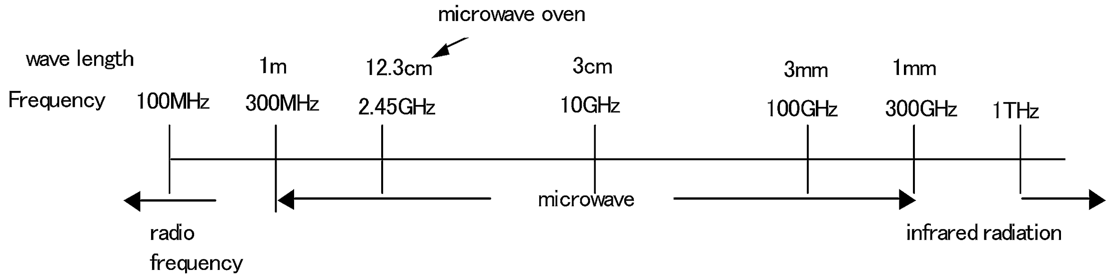

Fig. 1 Maicrowave frequency

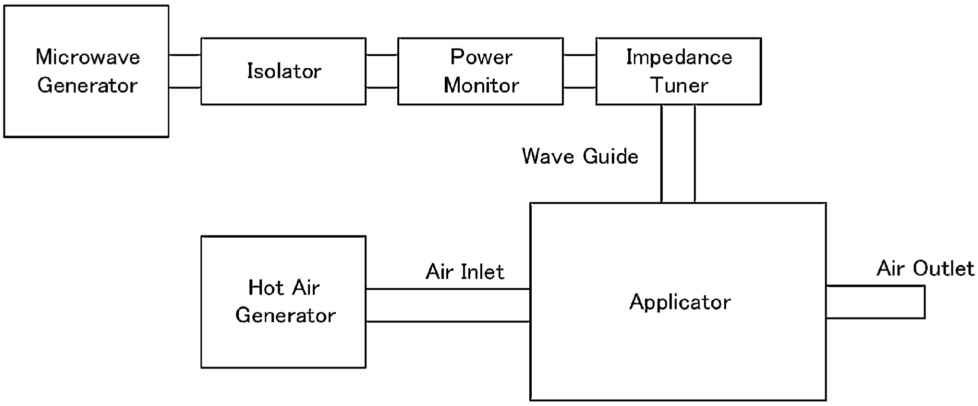

Fig. 3 Maicrowave drying system

Table 1 Chemical compositions of prepared alumina-magnesia castable

Component	Al_2O_3	MgO
	93mass%	5 mass%

Table 2 Chemical compositions of prepared alumina-spinel castable

Component	Ladle	RH
Al_2O_3	93 mass%	93 mass%
MgO	3 mass%	5 mass%

was used to measure the heat-up conditions of the interior of a large block using microwaves) and then poured into a metallic form measuring 500 mm × 500 mm × 300 mm. After the mixture was formed into a block on a vibrating table, it was cured for 24 hours. In order to measure the internal temperature of the refractory, an alumina protective tube 5 mm in OD and 3 mm in ID was embedded in the refractory during its formation, and K-thermocouples were installed in the protective tube when the refractory was dried. The measuring points were 25, 50, 75, 100, 150, 225, 250 and 275 mm from the top of the sample (300 mm in height). The refractory internal pressure was measured during drying using a copper tube (2 mm OD, 1 mm ID) embedded in the refractory during its formation. The measuring points were 75 mm, 150 mm and 225 mm from the top of the sample.

In addition to the above samples, blocks measuring 200 mm × 200 mm × 200 mm were prepared to measure the refractory's physical properties. They were subjected to drying by hot air and microwaves, as were the other samples.

3.3 Conditions for microwave drying

The alumina-magnesia refractory sample was installed in the applicator and dried using a microwave of 2 to 6 kW/t at an atmospheric temperature of 120℃ attained by feeding hot air into the applicator. To dry the alumina-spinel refractory sample, which was larger than the alumina-magnesia refractory sample, a 4-kW/t microwave was used at the same atmospheric temperature.

3.4 Measurement of physical properties after microwave drying

Concerning the alumina-magnesia monolithic refractory, a specimen of prescribed shape for measurement of physical properties was cut out by wet working from the sample that had already been subjected to microwave drying. After the specimen was dried at 110℃, it was subjected to measurement of bulk density, apparent porosity, flexural strength, compressive strength, elasticity, linear change and permeability in compliance with JIS. In addition, a specimen was cut out from the above sample for the rotating corrosion test. After the specimen was dried in the same way as mentioned above, its resistance to corrosion and slag penetration at 1,650℃ was evaluated using a composite slag of mass ratio 1:1 (C/S = 3.5) and ordinary steel (SS400) as eroding agents.

As for the alumina-spinel monolithic refractory, a specimen of prescribed shape was cut out by wet working from the 200 mm × 200 mm × 200 mm block that had already been dried using the microwave. After the specimen was dried at 110℃, it was subjected to measurement of bulk density, apparent porosity, flexural strength, elasticity, linear change and permeability.

4. Experimental Results and Considerations

4.1 Alumina-magnesia monolithic refractory

4.1.1 Microwave drying of alumina-magnesia monolithic refractory

Fig. 4 shows the changes in temperature and internal pressure of sample blocks with 5.6 mass% water (minimum water content) and 6.3 mass% water (maximum water content), respectively, when the blocks were dried by a microwave of 2 to 6 kW/t. It can be seen that when a 200-mm thick block is dried using a microwave, the entire block is dried almost uniformly, with minimal temperature difference. It can also be seen that the temperature of the central part (100 mm from top) of the block is slightly higher than that of the top and bottom of the block. This is a phenomenon unique to internal heating by microwaves.

Looking at the block's heat-up behavior, when the microwave output is low (2 kW/t) and the temperature is higher than 100℃, the heat-up rate decreases due to the latent heat of evaporation of water. With an increase in microwave output, the heat-up rate tends to increase. Since the internal pressure is actually the steam pressure, the point in time at which the internal pressure sharply declines can be regarded as the time when the free water has been completely evaporated out.

The drying times of alumina-magnesia blocks with 5.6 mass% water and 6.3 mass% water, respectively, are shown in **Tables 3 and 4**. Under the present experimental conditions, the drying time could be decreased by some 10 hours, from about 32 hours to 22 hours, by increasing the microwave output from 2 kW/t to 6 kW/t.

From the internal pressures measured while the microwave output was 4 kW/t and 6 kW/t, respectively, it was confirmed that the internal pressure measured during heat-up of the material nearly coincides with the measured equilibrium steam pressure that is determined by the temperature of the center (100 mm from top) of the block.

From Table 4, it can be seen that under the present experimental

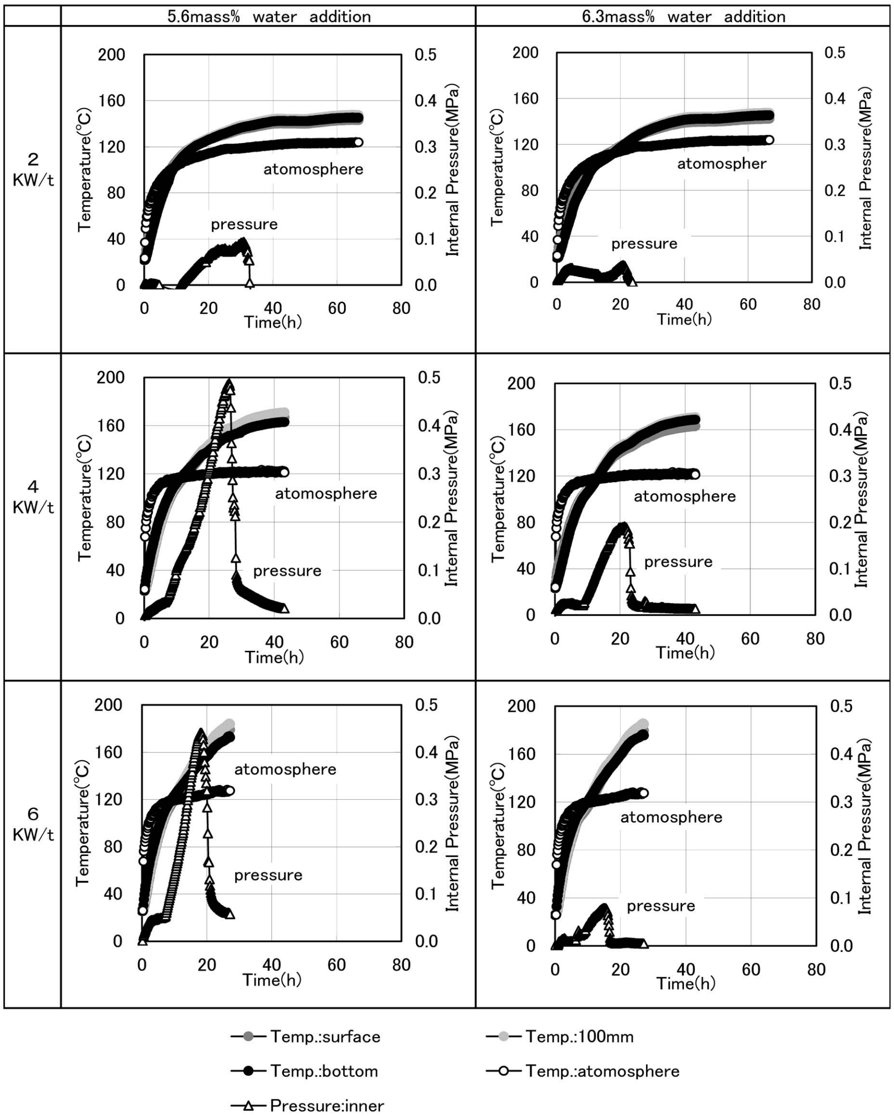

Fig. 4 Temperature and internal pressure in the blocks during microwave drying of alumina-magnesia monolothic refractories

Table 3 Drying time of alumina-magnesia castable with 5.6mass% water addition

Microwave power (kW/t)	2	4	6
Drying time (h)	32	28	22

Table 4 Drying time of alumina-magnesia castable with 6.3mass% water addition

Microwave power (kW/t)	2	4	6
Drying time (h)	23	23	17

conditions, the drying time could be decreased by some 6 hours, from about 23 hours to 17 hours, by increasing the microwave output from 4 kW/t to 6 kW/t. It can also be seen that as the amount of water added is increased, the drying time generally decreases. This is considered due to the permeability of refractory block described later. It is considered that with the increase in the amount of water added, more paths for mass transfer are created when the water evaporates. As a result, the absolute value of the internal pressure generated is smaller than when a small amount of water is added.

4.1.2 Material characteristics of alumina-magnesia monolithic refractory after being dried by microwave

Fig. 5 shows the changes in mechanical and chemical properties of alumina-magnesia refractory dried using a microwave when the amount of water added was varied. Each of the properties shown here, excepting corrosion resistance and slag penetration resistance, is the average value of a total of 12 test pieces—6 test pieces (40 mm × 40 mm × 40 mm) cut out from each of two samples (200 mm × 200 mm × 200 mm). For corrosion resistance and slag penetration resistance, the average amount of wear and the average amount of slag penetration, each obtained from three test pieces, are shown as an index. The smaller the index, the better each of these properties is.

There is a tendency that with the decrease in the amount of water added, the bulk density increases and the porosity decreases, that is, the refractory material becomes denser. Accordingly, the flexural and compressive strengths, and even the modulus of elasticity, tend to

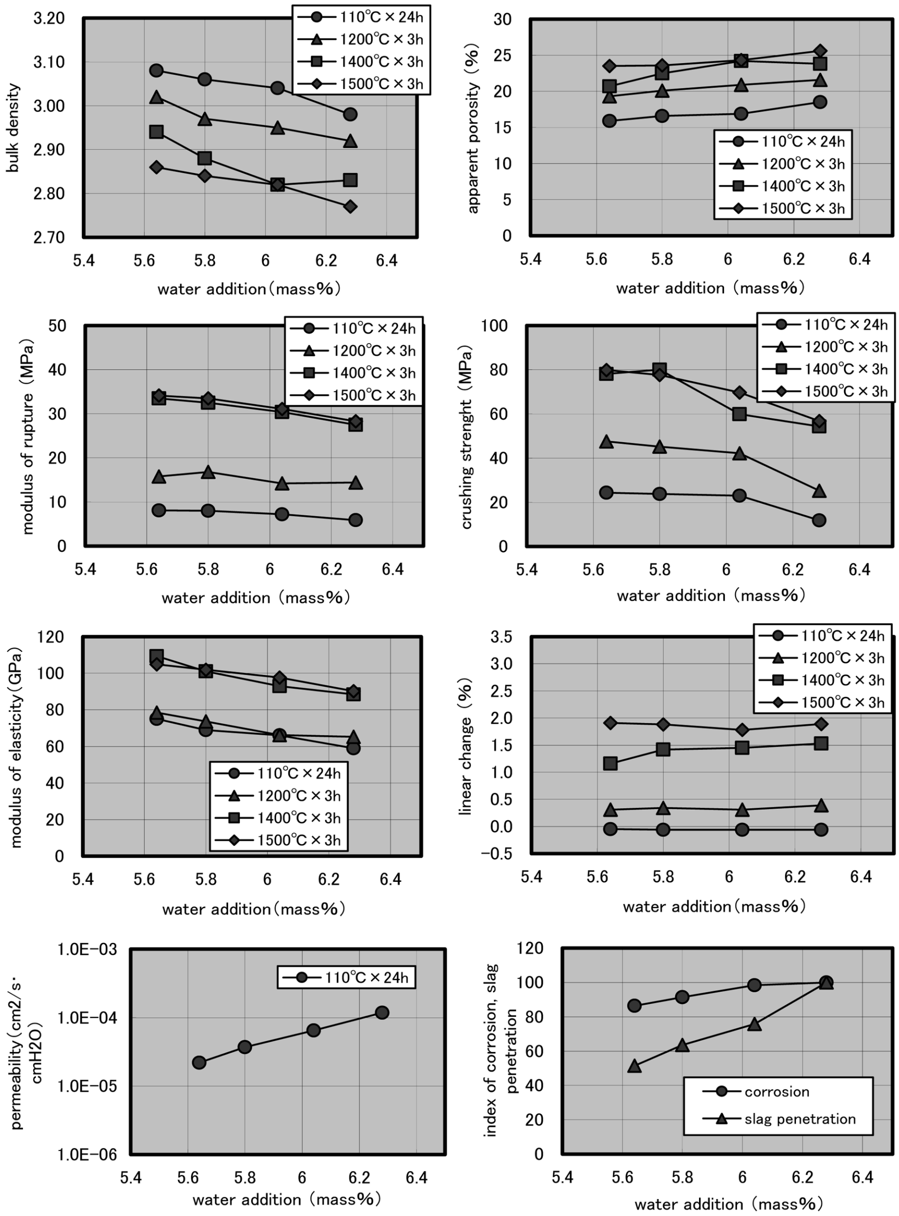

Fig. 5 Relationship between the water addition and some physical and chemical properties

increase.

It seems that the amount of water added has minimal influence on the rate of linear change of the refractory. Concerning the permeability, it decreases by one order of magnitude when the amount of water added is decreased from 6.3 mass% to 5.6 mass%. In this case, it is considered that the transfer of steam becomes difficult in the denser block. This is considered to account for the increase in drying time mentioned earlier.

As for the corrosion resistance, it improved by some 17% when the amount of water added was decreased from 6.3 mass% to 5.6 mass%. More strikingly, the slag penetration index decreased to about half. It was already known that making the material denser is an effective means of improving the corrosion resistance of refractory material. From the present experiment, it was newly discovered that from the standpoint of restraining slag penetration, reducing the permeability is most effective. From the experimental results described above, we found it possible to improve the corrosion resistance of monolithic refractory by reducing the amount of water added during refractory work, even without modifying the refractory's chemical composition.

4.2 Alumina-spinel monolithic refractory

4.2.1 Microwave drying of alumina-spinel monolithic refractory

Fig. 6 shows the changes in temperature and internal pressure measured during microwave drying of a 500 mm × 500 mm × 300 mm block of alumina-spinel refractory. The internal pressure is actually the steam pressure, and the point in time at which the internal pressure sharply drops indicates that the free water has completely evaporated out. In microwave drying, the drying of free water was completed in about 31 hours. As in the case of microwave drying of alumina-magnesia monolithic refractory with a microwave output of 4 kW/t or more discussed above, the phenomenon whereby the heat-up rate decreases due to the latent heat of water evaporation is

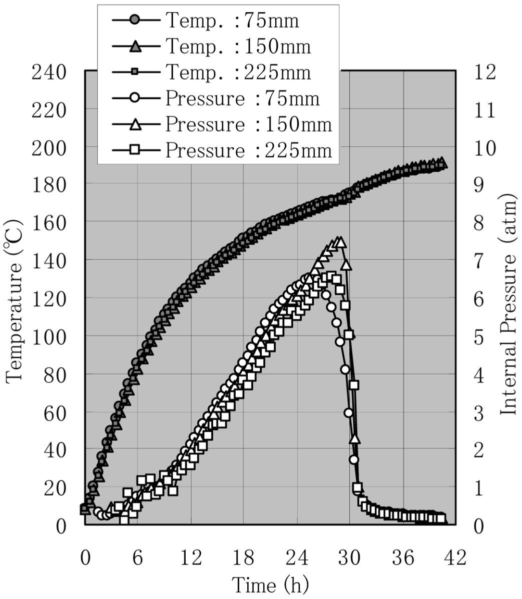

Fig. 6 Temperature and internal pressure in the blocks during microwave drying of alumina-spinel monolothic refractories

Table 5 Physical properties of alumina-spinel monolithic refractories

Bulk density	3.16
Apparent porosity (%)	11.1
Modulus of rupture (MPa)	-
Crushing strenght (MPa)	130
Modulus of elasticity (GPa)	133
Permeability ($m^2/s \cdot cmH_2O$)	3.4×10^{-6}

unclear and the difference between the surface temperature and the internal temperature is minimal even when the block dried by microwave is larger in size.

Probably the reason for this is that in microwave drying, which is based on internal heating, the inside of the refractory where the water exists is heated and the heat generated is transferred toward the surface. In other words, since the heat is transmitted from the high side toward the low side, it is considered that when the heat is transferred from the inside toward the surface, the surface temperature is lower than the internal temperature and that the surface temperature does not become higher than the internal temperature even after the drying process is completed. With respect to the narrow temperature distribution, it is considered due, at least in part, to the fact that water is capable of absorbing microwaves to such an extent that it is selectively heated by the microwaves.

4.2.2 Material characteristics of alumina-spinel monolithic refractory after being dried by microwave

Table 5 shows the physical properties of a test piece of alumina-spinel monolithic refractory cut out from a 200 mm × 200 mm × 200 mm block after microwave drying.

5. Status of Introduction of Hot Air & Microwave Drying Equipment at Nippon Steel

Table 6 shows the status of hot air & microwave drying equipment introduced at Nippon Steel. At present, a total of ten units are in operation at the company's works. They are now used not only to dry ladles, RH vessels and other large structures lined with monolithic refractory from the beginning, but also large pre-cast refractory parts, such as refractory blocks for tuyeres and impact blocks.

Table 6 Microwave drying equipment in Nippon Steel

Works	Plant	Frequency power tube	Total power
Muroran	Precast blocks	2.45GHz Magnetron	21kW
Kimitsu 1SMP	Ladle	2.45GHz Magnetron	80kW
Kimitsu 2SMP	Ladle	2.45GHz Magnetron	100kW
Kimitsu	RH	915MHz Magnetron	150kW
Nagoya	Ladle	2.45GHz Klystron	120kW
Nagoya	Precast blocks	915MHz Magnetron	50kW
Hirohata	Ladle	2.45GHz Klystron	45kW
Yawata	Precast blocks	2.45GHz Klystron	120kW
Oita	Ladle	2.45GHz Magnetron	100kW
Oita	RH	2.45GHz Magnetron	30kW

6. Conclusion

We studied the behavior of alumina-magnesia and alumina-spinel refractories used for ladles and RH vessels when they are dried by a combination of hot air and microwaves. As a result, it was found that the hot air & microwave drying process makes it possible to significantly narrow down the temperature distribution inside the refractory material during drying, regardless of the refractory size.

It was also found that in monolithic refractory work, when the amount of water added is decreased, the porosity decreases, the steam pressure generated inside the refractory rises, and the drying time increases despite the decreased amount of water added, but that the increased density of the refractory material improves the corrosion resistance appreciably and reduces the amount of slag penetration dramatically.

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