

大型超高温ジルコニア炉

Large-sized Zirconia Ultra-high Temperature Furnace (ZRF: Zircofurnace)

笹井 洋一^{*1}
Yoichi SASAI

奥原 潤一郎^{*2}
Junichiro OKUHARA

Abstract

The zirconia furnace is an electric furnace which enables heating to 2000°C in an oxidizing atmosphere. Our company is the only maker which has developed and markets practical zirconia furnace technologies. However, because the usable working dimensions of the zirconia furnace are small ($\phi 25\text{mm}$), its application had been limited to laboratory tests.

In recent years, there have been numerous needs for large-sized furnaces for use in calcining of products, but still issues remained in relation to temperature measurement accuracy and melting of the platinum wire terminal. Therefore, this company carried out various improvements, resulting in the development of higher-reliability furnaces. The developed technology is introduced in this report.

1. 緒言

ジルコニア炉は酸化雰囲気で2000°Cまで昇温可能な唯一の炉であり、これまでに大学関係を中心に30台以上の納入実績がある¹⁾。

Fig. 1にZRF-25の外観と炉内断面を、Table 1に仕様を示す。

昇温は、まず予熱ヒータでジルコニアヒータを通電可能温度1100°Cまで予熱した後、ジルコニアヒータへ通電させて、加熱室温度を2000°Cまで加熱させる。

本ジルコニア炉は、従来、温度測定器の問題から加熱室外部にB熱電対（測定温度max.1800°C）を加熱室内側と外側の温度差が300°C発生する位置（オフセット幅300°C）に挿入し、制御を行っていた。この時、次項2に示す問題が発生したが、各種改善を行い、より信頼性の高い炉ができたので紹介する。

1. Introduction

The zirconia furnace is the only furnace which enables heating to 2000°C in an oxidizing atmosphere. To date, Our company has delivered more than 30 of these furnaces, mainly to university-related clients.¹⁾

Fig.1 shows the external appearance of the ZRF-25 Zircofurnace and a cross-section of the furnace interior. Table 1 shows the specifications of the Zircofurnace.

In heating, first, the furnace is preheated using the preheater to a temperature of 1100°C, at which it is possible to pass a current through the zirconia heater. The heating chamber temperature is then increased to 2000°C by passing a current through the zirconia heater.

In view of the operating method for the temperature measurement device, conventionally, this zirconia furnace had been controlled by inserting a type B thermocouple (measurement temperature: maximum 1800°C) in the outside

^{*1} 技術研究所 第2研究グループ チームリーダー Installation Equipment Team Manager, Unshaped Refractories R&D Group, Research Center

^{*2} 技術研究所 第2研究グループ 主任研究員 Managing Research Engineer, Unshaped Refractories R&D Group, Research Center

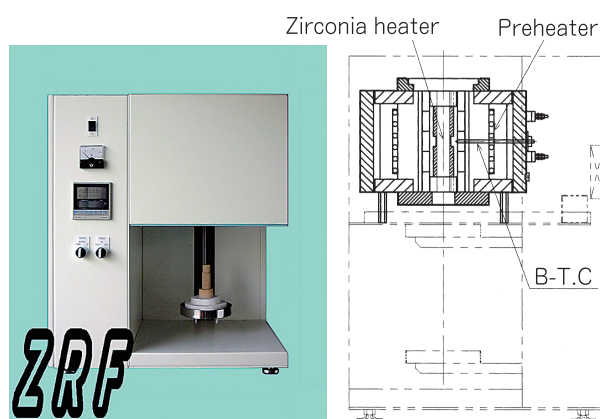


Fig. 1 Appearance and schematic diagram of ZRF-25 furnace.

Table 1 Specifications of Zircofurnace (ZRF)

Model	ZRF-25	ZRF-40	ZRF-120
Usable working dimensions (mm)	$\phi 25 \times 20H$	$\phi 40 \times 40H$	$\square 120 \times 120H$
Max. operating temperature	2100°C	2000°C	2000°C
Normal operating temperature	2000°C	1800°C	1800°C
Chamber & controller dimensions (mm)	700H $\times$ 600W $\times$ 500L		700H $\times$ 650W $\times$ 550L
Weight	100kg		160kg
Power	AC200V, 30A		
Heating capacity	5°C/min		
Heating atmosphere	Air		

2. 従前の問題点

- ① B熱電対の挿入長さが不足すると、炉内温度に対して制御用熱電対の反応が鈍く、ジルコニアヒータ自身に過電流が流れ、このため白金線が溶断しやすい。また、炉内温度が最高使用温度をはるかに超えてしまうので、ジルコニアヒータ寿命が低下。
- ② B熱電対を挿入しすぎると、炉内温度が目的の温度まで昇温できない。
- ③ ライニング構成断熱材の伝熱により、白金線端子部が過熱し、端子部が溶断しやすい。

part of the heating chamber at a position where the temperature difference between the inner side and outer side of the heating chamber was 300°C (offset width: 300°C). With this method, the problems mentioned in the following section 2 arose. However, in the present work, it was possible to develop a higher-reliability furnace by implementing various improvements. The developed technology is introduced in this report.

2. Problems with Conventional Technology

- ① If the insertion length of the type B thermocouple is inadequate, the response of the thermocouple for controlling the furnace temperature will be slow, resulting in over-current in the zirconia heater itself. For this reason, the platinum wire is prone to melting. Furthermore, since the furnace temperature tends to largely exceed the maximum operating temperature, the life of the zirconia heater will be reduced.
- ② If the type B thermocouple is inserted too deeply, the furnace will not heat up to its normal operating temperature of 2000°C.
- ③ The terminal part of the platinum wire tends to overheat due to the heat conduction of the heat-insulating material for lining, and as a result, the terminal part is also prone to melting.

3. Improvement Method

With the ZRF-25, a decision was made to improve the problems in the above section 2.

First, in order to improve temperature measurement accuracy, use of a radiation pyrometer in place of the type B thermocouple was studied.

3. 1 Improvement of temperature measurement accuracy

(1) Study of adoption of radiation pyrometer

The conventional radiation pyrometer had not reached practical application at the time of development of the ZRF-25 because it was necessary to open a large hole for use in measurements, which resulted in increased heat loss. Therefore, the furnace temperature had been controlled using a type B thermocouple, which is inexpensive and enables measurement with little heat loss.

Table 2 Comparison of radiation pyrometers

Model	Appearance	Measuring hole (mm)	Radiation heat loss (Requisite power ratio)
Conventional type		$\phi 30$	Approx.50%
Fiber type		$\phi 6$	Approx.2%

3. 改善手法

ZRF-25について、前項2の問題点の改善を図ることとした。

まず、温度測定精度を向上させるために、B熱電対に代わる放射温度計を検討した。

3.1 温度測定精度の向上

(1) 放射温度計の採用検討

従来の放射温度計は、測定用に大きな孔を開ける必要があり、放熱ロスが大きくなるので、当時は実用化には至っておらず、低コストで熱ロスが小さくて済むB熱電対を用いて制御していた。

従来式と超小型放射温度計の比較をTable 2に示す。

超小型放射温度計を用いた場合、測定孔径を非常に小さくできるので、放散熱ロスを小さくすることができ、昇温への悪影響は少ないと判断し、実用化を試みた。

なお、温度測定は、ジルコニアヒータ発熱部に直接孔を開けて行えないため、ジルコニアヒータ外面を測定することとした。さらに、炉材の熱膨張やファイバのヘタリにより炉材が動いてしまい、測定孔が貫通しなくなることが懸念されるので、測定孔を確保するため、アルミナ質パイプを通すことにした (Fig. 2)。

(2) 発熱部断熱強化

放射温度計で精度良く測定するためには、ジルコニアヒータ内・外面の温度差ができるだけ小さいことが前提となる。

A comparison of the conventional type and an ultra-compact fiber type radiation pyrometer is shown in Table 2.

Because the diameter of the measuring hole can be reduced to an extremely small size when the fiber type is used, it was judged that heat diffusion loss can be reduced and the negative effect on heating-up will be slight. Therefore, practical application was attempted.

Because it is not possible to open a hole directly in the heating zone of the zirconia heater, a decision was made to perform temperature measurements by measuring the outer surface of the heater. As there was also concern that penetration by the measuring hole might be lost due to movement of the furnace material (zirconia brick) resulting from the thermal expansion of the bricks and/or deterioration of the fiber, the fiber was passed through an alumina pipe in order to secure a measuring hole (Fig.2).

(2) Improvement of heat insulation of heating zone

For high accuracy measurement with a radiation pyrometer, minimization of the temperature difference between the inner and outer sides of the zirconia heater is a precondition.

As shown in Fig.2 (Fig.4b), when the fiber was inserted in the periphery of the zirconia material of the heating zone, and the upper and lower spaces were sealed with zirconia material, the temperature difference between the inside and outside of the zirconia heater could be reduced approximately 20°C in comparison with the conventional lining.

On the other hand, before improvement,

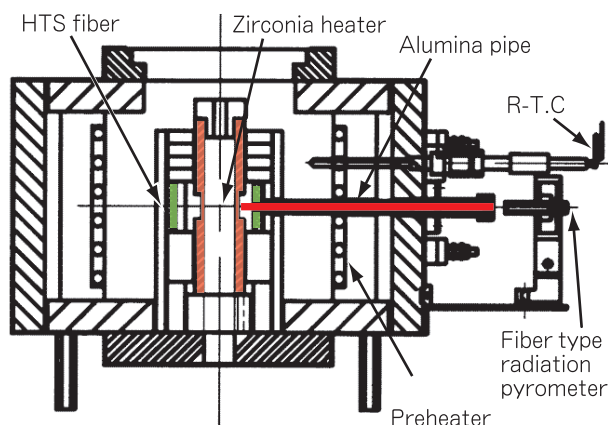


Fig. 2 New temperature measurement method for ZRF-25 furnace.

Fig. 2(Fig. 4b)のように、発熱部ジルコニア炉材の周囲にファイバを入れ、上下の空間を炉材でシールすると、従来のライニングに対し、ジルコニアヒータ内・外面の温度差が約20℃小さくなった。

一方、改善前の白金線端子部表面温度は、炉内2000℃の時に1740℃となっており、溶断温度1800℃近くまで過熱されていた。これは、ジルコニアヒータ発熱部からの伝熱、輻射および白金線の抵抗加熱によるものであり、発熱部を断熱強化することで熱流束が約20%減少することが計算により判った。

(3) 放射率の決定

放射温度計で物質表面の実態温度を測定しようとする、物質の種類、温度域によって、放射率が変化してしまう。よって、放射率の値を実態温度に合うように設定する必要がある。

そこで、温度ごとに光高温計で炉内実温度と外面温度を測定し、同時に放射温度計の放射率を変化させ、測定表示値を炉内実温度に合わせる手法により、放射率を求めた。

(4) 新規制御システム

従来システムでは、予熱ヒータからジルコニアヒータへの制御切り替わり時、予熱ヒータ制御時の出力信号をそのまま引き継ぐので、ジルコニアヒータが異常出力になりやすい欠点があったが、Fig. 3に示す新システムでは、予熱ヒータとジルコニアヒータは、独立制御されているので、制御

the surface temperature of the platinum wire terminal had reached 1740℃ when the furnace temperature was 2000℃, and the terminal had overheated to close to the melting temperature of platinum (approx. 1800℃). This was caused by heat conduction and radiation from the heating zone of the zirconia heater and the resistance heating of the platinum wire itself. From calculations, it was found that thermal flux is reduced by approximately 20% by strengthening the heat insulation of the heating zone.

(3) Determination of emissivity

As emissivity will differ depending on the type of substance and the temperature region, when the actual temperature of the surface of a substance is measured by a radiation pyrometer, it is necessary to set the value of emissivity corresponding to the actual temperature.

Therefore, the actual temperature inside outside of the furnace was measured by an optical pyrometer for several conditions, and simultaneously, the measured value displayed by the radiation pyrometer was matched with the actual temperature in the furnace by changing the emissivity of the radiation pyrometer.

(4) New control system

The conventional system had the drawback that the output from the zirconia heater easily became abnormal because the output signals during preheating heater control were also used as the signals for controlling zirconia heater when control was switched from the preheating heater to the zirconia heater. However, in the new system shown in Fig. 3, control switching is extremely smooth because the preheater and zirconia heater are controlled independently. Furthermore, measured values and setting values up to 2000℃ can be displayed on the program temperature controller.

3. 2 Measures against melting of platinum wire terminal

(1) Improvement in sealing of heating zone

As a further measure against trouble involving melting of the platinum wire, an overheating prevention measure was applied to the terminal part.

Before the improvement, there was a space between the zirconia brick and the zirconia heater, and the zirconia heater terminal had overheated due to radiation and gas rise from

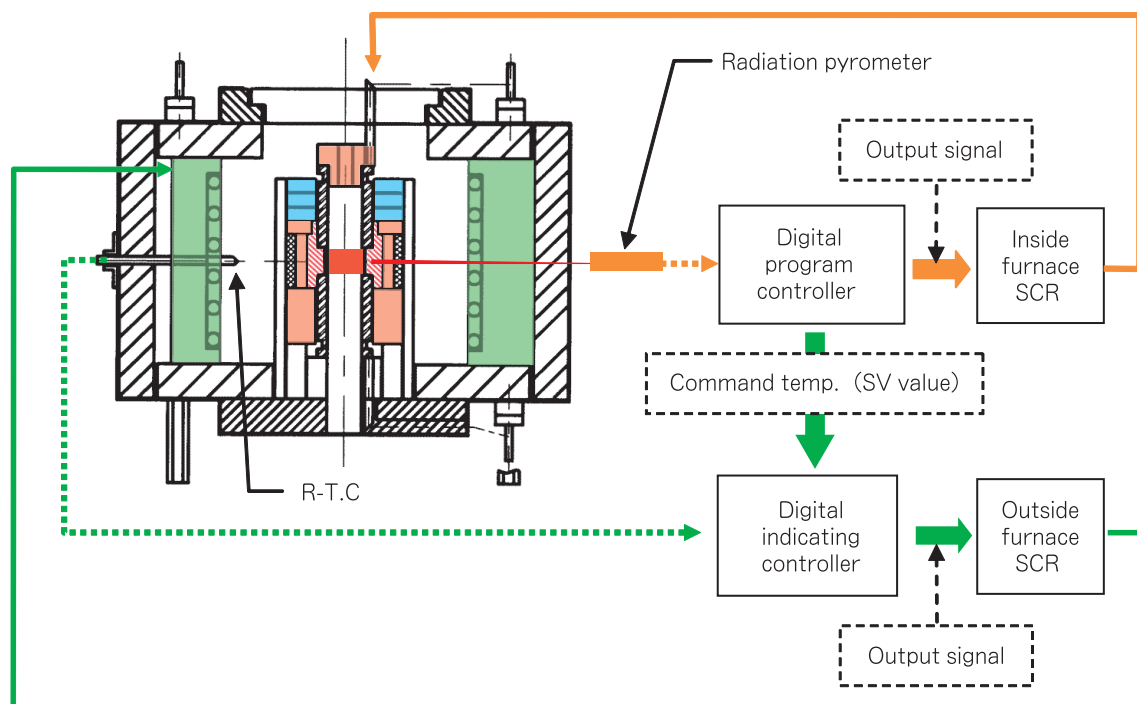


Fig. 3 New temperature control system for ZRF-25 furnace.

切替が非常にスムーズになった。また、プログラム温度調節計の設定値と測定値も2000℃表示が可能となった。

3. 2 白金線端子部の溶断対策

(1) 発熱部シール性向上

さらなる白金線溶断トラブル対策として端子部の過熱防止対策を行った。

改善前、ジルコニアヒータ端子部は、ジルコニア炉材とジルコニアヒータの間に空間があり、発熱部からの輻射およびガス上昇により過熱されていた (Fig. 4a)。そこで、白金線端子部の過熱防止対策として、発熱部の上下をジルコニア炉材でシールして発熱部の熱が端子部に悪影響を及ぼさないようにした (Fig. 4b)。

(2) 端子部絶縁対策

従来ライニングでは、ジルコニアヒータ白金線端子部がジルコニア炉材で囲われているため、両者の間で導通し、端子部がスパーク溶損することがあった。

そこで、ジルコニア炉材をMgO絶縁板に置き換え、白金線端子部とジルコニア炉材の間を絶縁し

the heating zone (Fig.4a). Therefore, as a measure to prevent overheating of the platinum wire terminal, the upper and lower parts of the heating zone were sealed with zirconia brick material to prevent the adverse effect of the heat of the heating zone on the terminal part (Fig.4b).

(2) Electrical insulation measure for terminal part

With the conventional lining, the platinum wire terminal of the zirconia heater is surrounded by zirconia brick. As a result, there were cases in which spark erosion of the terminal occurred due to electric conduction between the two materials.

Therefore, the zirconia brick material was replaced with MgO electrical insulating plates, thereby providing electrical insulation between the platinum wire terminal and the zirconia brick (Fig.4b).

4. Results of Improvement (ZRF-25)

Table 3 shows a comparison of the results of heating tests before and after improvement.

In the ZRF-25, the above-mentioned improve-

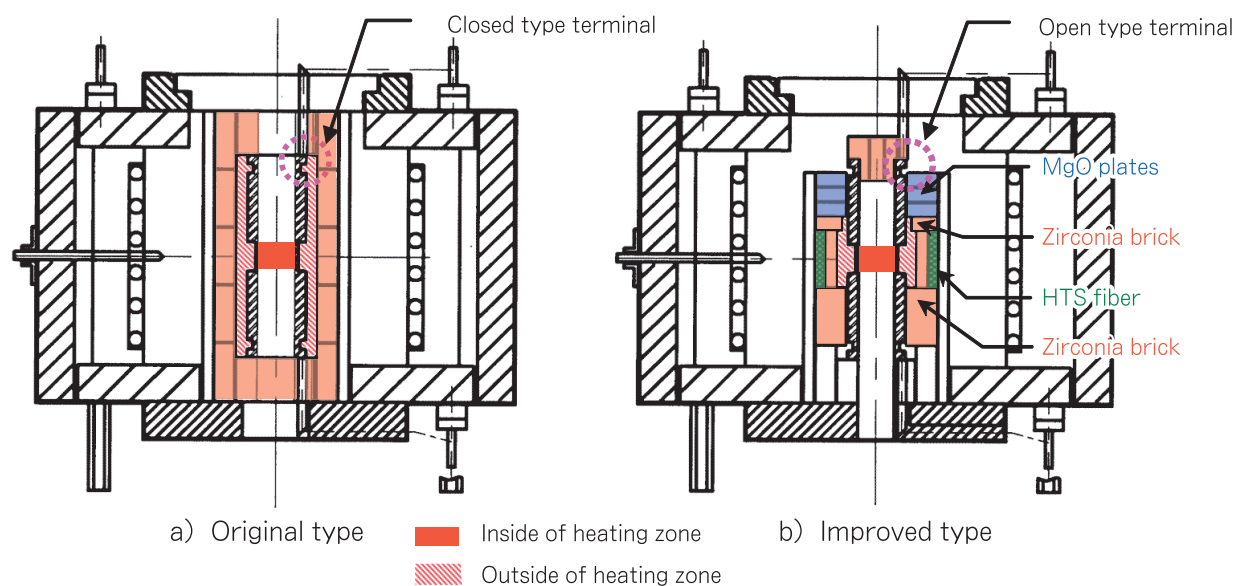


Fig. 4 Schematic diagram of ZRF-25 furnace.

Table 3 Results of heating test of ZRF-25 furnace

Item	Original type	Improved type	Remarks
Control temperature (actual temp. 2000°C)	Setting temp. 1700°C	Setting temp. 2000°C	
Measurement accuracy (actual temp. 2000°C)	Vary widely according to setting position of B-T.C. <Standard value> +10mm; 1800°C - 5mm; 2200°C	2000°C+10°C -20°C	
Temperature controllability	Response varies according to controller output (hunting)	Steady response (non-hunting)	
Heating capacity	Max 5°C/min	Max 7.5°C/min	
Current value	Max 20A	Max 17A	Actual temp. 2000°C
Surface temp. of terminal end	1740°C	1470°C	Pt melting temp. Approx. 1800°C
Lining design	Risk of spark erosion	Improved stability	
Life (number of heats)	3~20 heats	95 heats	

た (Fig. 4b)。

4. 改善結果 (ZRF - 25)

Table 3に改善前後の結果比較を示す。

ZRF - 25については、上述の温度測定精度の向上およびジルコニアヒータ加熱室の断熱強化対策により、電流値も安定し、発熱体の寿命延長に繋がった。

温度測定精度は、2000°C時の炉内温度について、放射温度計の表示温度と光高温計で測った炉内実

ment of temperature measurement accuracy and measure to strengthen the heat insulation of the the zirconia heating chamber resulted in stability of the current value and greatly extended the life of the heating element.

Temperature measurement accuracy was improved to +10°C, -20°C at a temperature of 2000°C, which was confirmed by a comparison of the temperature readings of the radiation pyrometer and the actual furnace temperature measured by an optical pyrometer. Furthermore, it was possible to display the actual

温度を比較確認したが、 $+10^{\circ}\text{C}$ 、 -20°C で収まった。さらに、炉内実温度 2000°C がプログラム温度調節計に表示可能となった。

また、断熱強化後の電流値は、炉内 2000°C 時に $20\text{A}\rightarrow 17\text{A}$ と約15%減、白金端子表面温度は、 $1740^{\circ}\text{C}\rightarrow 1470^{\circ}\text{C}$ に低下し、安全率を向上させることができた。昇温回数も前述のトラブルを回避し、95回をクリアした。

なお、 $\phi 25\text{mm}$ の発熱体については、白金線端子部を発熱部からさらに離してやれば、端子部の過熱を抑えることができるとわれ、さらなる安全率向上が期待できる。また同時に、発熱体コストの大部分を占める白金線使用量を減らすことも可能と思われる。

5. 新規測定方法の大型炉への適用

従来、 $\phi 40\text{mm}$ や $\square 120\text{mm}$ 発熱体についても、 $\phi 25\text{mm}$ と同様にB熱電対をオフセットした位置に配置し制御していた。ただし、下記の問題があった。

- ①B熱電対は加熱室内の上部に配置されており、そのため 1800°C までしか測定できなかった。
- ②また、B熱電対を上部に配置していることから、光高温計での実温度測定が困難であった。

これに対し、 $\phi 40\text{mm}$ 発熱体について、端子部側面に測定用の孔を開けて、Fig. 5のように超小型放射温度計を利用し、温度制御を試みた。放射率は、 $\phi 25\text{mm}$ 発熱体で決定した数値をそのまま使用した。また、天井に確認用のB熱電対を取り付けた。

昇温は放射温度計で 2000°C まで行い、B熱電対を用いて所定の位置で測定しようとしたところ、所定の位置に行くまでにB熱電対は溶断した。 $\phi 40\text{mm}$ 発熱体については、光高温計での実温度測定は困難であるが、ほぼ 2000°C まで昇温し、制御できたと思われる。なお、制御性については、 $\phi 25\text{mm}$ 発熱体と同様に良好であった。

今後、 $\square 120\text{mm}$ 発熱体についても、同様のテストを実施予定であり、 $\square 120\text{mm}$ については光高温計で実温度を測定可能なスペースがあるので、検証を行う予定である。

furnace temperature of 2000°C on the program temperature controller.

The current value after strengthening of the heat insulation was reduced by approximately 15%, from 20A to 17A, at a furnace temperature of 2000°C . As a result, the surface temperature at the platinum wire terminal was reduced from 1740°C to 1470°C , thereby increasing the safety factor. Because it was possible to avoid the above-mentioned trouble, a furnace life (number of heats) of 95 heats was successfully achieved.

Regarding the $\phi 25\text{mm}$ zirconia heater, if the platinum terminal can be separated further from the heating zone, it would seem that overheating of the terminal can be avoided, and a further improvement in the safety factor can be expected. At the same time, it would also seem possible to reduce the use of platinum wire, which accounts for the largest part of the cost of the heating element.

5. Application of New Measurement Method to Large-sized Furnaces

Conventionally, the $\phi 40\text{mm}$ and $\square 120\text{mm}$ heaters had been controlled by arranging a type B thermocouple at an offset position, in the same manner as with the $\phi 25\text{mm}$ device. However, this had the following problems:

- ①Because the type B thermocouple was arranged at the top part in the heating chamber, it was only possible to measure temperatures up to 1800°C .
- ②Furthermore, because the type B thermocouple was arranged in the top part, measurements of the actual temperature by an optical pyrometer were difficult.

To solve these problems, with the $\phi 40\text{mm}$ heating device, a measuring hole was made at the side of the terminal, and temperature control was attempted using an ultra-compact fiber type radiation pyrometer, as shown in Fig.5. For emissivity, the value decided with the $\phi 25\text{mm}$ heater was used without modification. In addition, a type B thermocouple for confirmation measurements was installed in the furnace roof.

When heating-up was performed to 2000°C with the radiation pyrometer and the temperature was measured at the specified position using the type B thermocouple, the thermocouple had

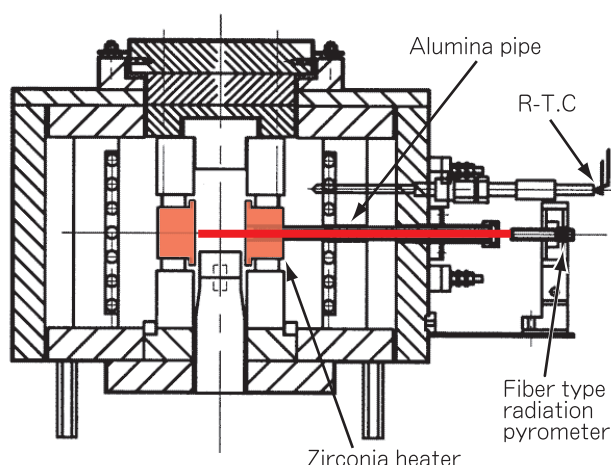


Fig. 5 New temperature measurement method for ZRF-40 furnace.

6. 今後の課題

現在、 $\phi 40\text{mm}$ や $\square 120\text{mm}$ の発熱体は、発熱部の近くに白金線を埋め込む形を採用している。これは、加工上の問題から、端子部を外部に出すことが困難なためである。

このため、 $\phi 40\text{mm}$ や $\square 120\text{mm}$ の発熱体は、白金線端子部が過熱されやすいので、若干冷却エアを入れて昇温している。しかし、この方法については、電流値増加の観点から、あまり好ましい方法ではない。現在、端子部の温度ができるだけ上がらないように、端子部を外部に出す方法を模索中である。

また、 $\square 120\text{mm}$ 発熱体の形状については、形状が大きいため、熱応力の問題が発生しており、この熱応力を緩和する形状の見直しを行う必要があるため、さらなる改善を進めていく所存である。

melted before reaching the specified position. Although, for the $\phi 40\text{mm}$ heater, measurement of the actual temperature by the optical pyrometer was impossible, it appeared that the device could be heated/controlled at temperatures up to substantially 2000°C . Controllability was also satisfactory, as with the $\phi 25\text{mm}$ heater.

Similar tests with the $\square 120\text{mm}$ heater are scheduled for the future. Because space for measurement of the actual temperature by an optical pyrometer is available with the $\square 120\text{mm}$ type, verification of the actual temperature is planned.

6. Future Issues

At present, a method in which a platinum wire is embedded close to the heating zone has been adopted with the $\phi 40\text{mm}$ and $\square 120\text{mm}$ heaters. This method is used because it is difficult to manufacture a device in which the terminal extends to the outside due to machining-related problems.

Because the terminal of the platinum wire easily overheats in the $\phi 40\text{mm}$ and $\square 120\text{mm}$ heaters with this design, a small amount of cooling air is introduced during heating-up. However, from the viewpoint of the increased current value, this is hardly a satisfactory method. At present, the authors are searching for a method in which the terminal extends to the outside so as to minimize the increase in the temperature of the terminal.

With the $\square 120\text{mm}$ heater, the problem of thermal stress arises due to its large shape. As it is necessary to conduct a review of the shape so as to alleviate this thermal stress, the authors intend to make further improvements in this device.

Reference

- 1) Y. Sasai ; Shinagawa Technical Report, 49, 135-138 (2005)