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Investigation of Furnace Uniformity and its Effect on High-Temperature Fixed-Point Performance

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Abstract A large-area furnace BB3500YY was designed and built at the VNIIOFI as a furnace for high-temperature metal (carbide)–carbon (M(C)–C) eutectic fixed points and was then investigated at the NMIJ. The dependence of the temperature uniformity of the furnace on various heater and cell holder arrangements was investigated. After making some improvements, the temperature of the central part of the furnace was uniform to within 2 K over a length of 40 mm—the length of the fixed-point cell—at a temperature of 2,500°C. With this furnace, the melting plateaux of Re–C and TiC–C were shown to be better than those observed in other furnaces. For instance, a Re–C cell showed melting plateaux with a 0.1 K melting range and a duration of about 40 min. Furthermore, to verify the capability of the furnace to fill cells, one Re–C and one TiC–C cell were made using the BB3500YY. The cells were then compared to a Re–C cell made in a Nagano furnace and a TiC–C cell filled in a BB3200pg furnace. The agreement in plateau shapes demonstrates the capability of the BB3500YY furnace to also function as a filling furnace.

Keywords Eutectic · Fixed point · Furnace · High temperatures · Melting · Rhenium · Titanium

B. Khlevnoy and K. Anhalt are guest scientists at NMIJ at the time of investigation.

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1 Introduction

High-temperature fixed points (HTFP) based on metal-carbon systems (M(C)–C fixed points) have attracted the attention of many researchers and hold the promise to take an important role in radiometry and radiation thermometry in the near future [1,2]. These new HTFPs need furnaces with working temperatures that exceed 2,500°C (for Re–C) and 2,800°C (for TiC–C). The quality of a furnace, especially its temperature uniformity, is very critical for the reproducibility and plateau quality of the fixed points [3,4]. Three types of furnaces are conventionally used for the highest temperature M(C)–C fixed points: Thermogage, Nagano, and the VNIIOFI-made BB3200pg/BB3500 [5]. The last one was originally designed as a variable-temperature blackbody for spectral irradiance and later adapted as a furnace for HTFPs. A new furnace, BB3500YY, was designed and manufactured at the VNIIOFI especially for HTFP applications and then investigated at the NMIJ. The present paper describes the furnace and the results of investigations, which were presented in part at the NEWRAD 2005 conference [6,7].

2 Furnace Design

2.1 General Design

The BB3500YY furnace, dedicated to HTFP fabrication and plateau realization, was designed for a maximum working temperature of about 3,200°C and improved temperature uniformity. The furnace accommodates a fixed-point cell of 24 mm outer diameter and is similar to, but with a larger heater than, the pyrographite (PG) blackbody BB3200pg [8].

A photo and cross section of the BB3500YY are shown in Fig. 1. It has a water-cooled housing consisting of a main cylindrical part and front and rear flanges. The resistance-type cylindrical heater of the furnace consists of a set of PG rings compressed by a spring between fixed front and movable rear electrodes. The spring mechanism, which compensates the heater thermal expansion, and the movable graphite electrode are assembled on the rear flange of the housing, while the front copper flange serves as the front electrode. The heater is surrounded by a thermoshield made of carbon cloth wrapped around a cylindrical graphite bobbin.

A fixed-point cell can be placed inside the heater in the center of the furnace by means of a holder. This part of the furnace should have a temperature distribution as uniform as possible to provide conditions for uniform melting of the fixed-point material, thereby enabling a flat melting plateau. Some features of the PG furnace help to meet this requirement. First of all, the flexibility in the ring ordering provides a means to adjust the temperature distribution. It is obvious that the rings located closer to the electrodes lose more heat due to thermal conduction and radiation, and therefore tend to become cooler than those closer to the center. In order to compensate for these losses and to improve the temperature distribution, the rings are placed in order according to their electrical resistance: the resistance of the rings is lowest for the rings in the center of the furnace and gradually increases towards the ends of the heater. The thermal homogeneity is further improved by graphite baffles that separate

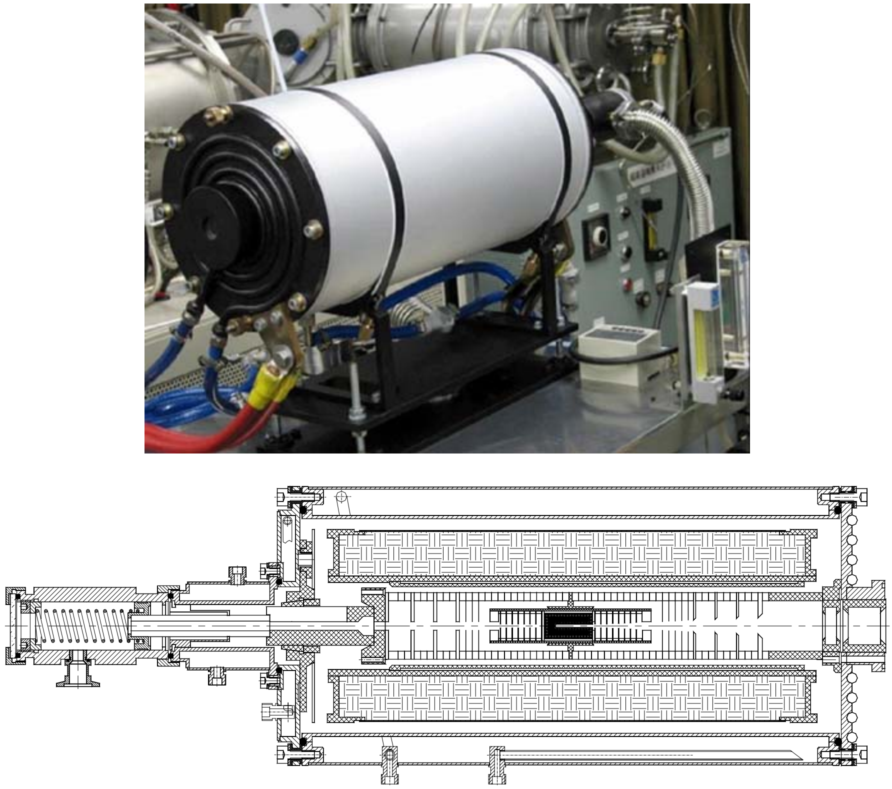

Fig. 1 Photo and cross section of the BB3500YY furnace

the central volume of the furnace. From a practical point of view, the ring-set design makes PG furnaces very flexible; numerous baffles and a cell holder can be easily placed at any position between the rings, and changing the ring order can change the temperature distribution.

Normally, the BB3500YY is operated in an argon atmosphere without any window in front of its opening, but it can also be used in vacuum with a quartz window. It has a rear radiation channel that can be used for a monitor/control pyrometer or thermocouple.

2.2 Adaptation Based on Previous Designs

In comparison with previous PG furnaces, the BB3500YY has a longer thermoshield and a longer and larger set of PG rings, with lengths 455 and 355 mm, respectively (instead of 370 and 290 mm for the BB3200pg), and a ring inner diameter of 47 mm (compared to 37 mm for the BB3200pg). The length of the ring set can be increased to 400 mm if necessary. The increased length enables the central part of the furnace to be further from the cold ends of the heater, which makes its temperature relatively more uniform. The increased inner diameter of the rings also improves the temperature uniformity because of better conditions for radiative heat exchange. The larger space between the cell and the rings allows placement of additional screens for further improvement of thermal uniformity.

2.3 Modifications to Improve Furnace Performance

Originally, the BB3500YY had 8 PG baffles of 4-mm thickness, four in the front part and four in the rear. The cell holder was originally made from graphite, 80 mm in length and 30 mm in outer diameter, and designed to hold a cell 45 mm in length and 24 mm in diameter. In order to improve the temperature uniformity, more baffles made from thin (about 0.1 mm) PG sheets were later added between the holder and the nearest 4-mm thick baffles. Baffles of the same material were placed inside the holder as well, in front of and behind the cell. Later, the graphite holder was replaced by one made of 150-mm carbon-fiber-reinforced carbon composite (C/C) tube similar to that used in the Nagano VR10-A23 furnaces [3]. Figure 1 shows the arrangement with the C/C-tube holder.

A feedback optical system was introduced into the BB3500YY. It is based on a commercially available optical-fiber pyrometer and a temperature controller. The pyrometer looks inside the furnace through its rear channel, viewing the baffle behind the cell. The system provides convenient temperature control of the furnace.

3 Measurement of the Heater Ring Resistance

In the following sections, investigations are described that show the significant influence of the heating efficiency of PG rings on the temperature uniformity of the heater. Firstly, in this section, a method is presented to measure the rings' electrical resistances, which determine the heating efficiency.

The resistance of the rings used for the furnace construction is not controlled at manufacture. During furnace construction, all the rings are aged at a temperature of about 3,200°C for a few hours, machined to the final dimensions, and then their electrical resistance is measured at room temperature. To be specific, the value we are interested in is not the resistance of each individual ring, but the resistance per unit of ring thickness. Therefore, when we refer to ring resistance throughout this paper we actually mean "resistance/ring thickness." To measure this characteristic property, each PG ring was pressed between two water-cooled metal disks that served as electrodes to pass a current of 20 A through the ring. Then, a two-needle probe with 1.8 mm spacing between the needles was used to touch the cylindrical outer surface of the ring to measure the voltage drop across the needles, with the plane of the needles perpendicular to the plane of the ring. The facility is shown in Fig. 2. For each ring, the voltage is measured at several different positions and the average value is then used as an indicator of the resistance/ring thickness.

Two nearly identical heaters were prepared with a ring order resulting in the heater-resistance profile shown in Fig. 3. The ring resistance remained relatively stable while the furnace was operated in argon. However, there was a great change when one of the

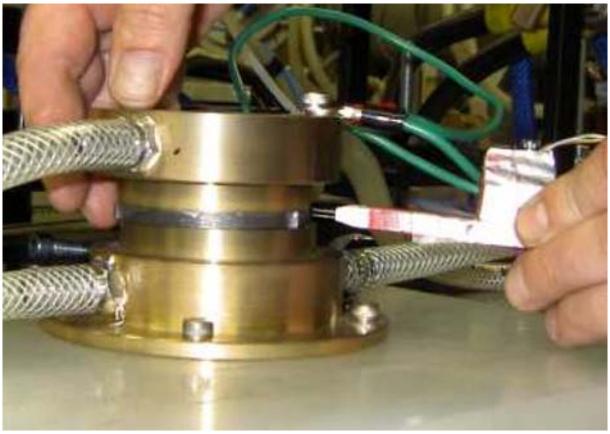

Fig. 2 Facility for PG rings' resistance measurement

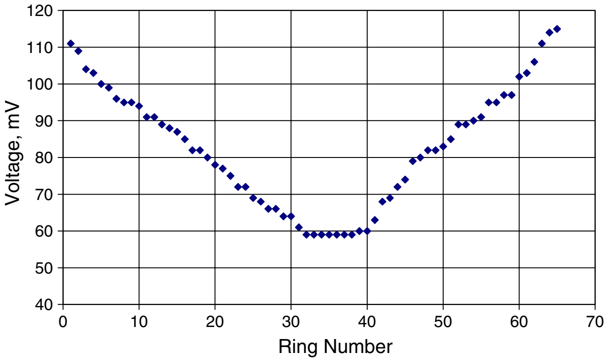

Fig. 3 Original resistance profile of the BB3500YY heater, presented as the measured voltage drop across a 1.8 mm length of heater with 20 A current at room temperature. First ring is nearest to the front flange

heaters was operated in vacuum at a temperature of about 2,900°C for a few hours. After that, it was found that about half of the rings (those closer to the ends of the heater) became covered with a thin layer of PG. The covered rings can be seen in Fig. 4 and identified by the lighter color. Their resistance was measured and found to be about half the value measured prior to vacuum operation. PG is a highly anisotropic material having an electrical conductivity much higher in the plane of the deposited layers than perpendicular to the layers. Therefore, the deposited layer had shunted the rings underneath. However, the ring resistance could be recovered by physically removing the deposited layer. For instance, one of the rings showed a voltage drop of 120, 50, and 115 mV, respectively, before vacuum treatment, after vacuum treatment, and after removing the deposited layer.

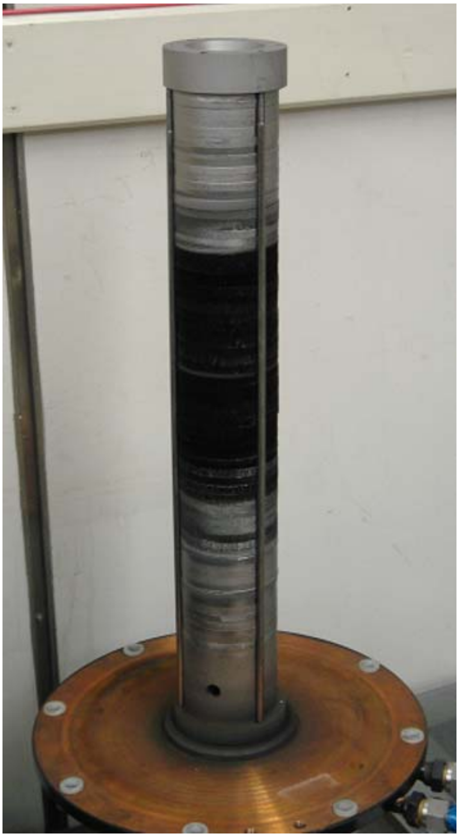

Fig. 4 View of the heater after operating the BB3500YY in vacuum at 2,900°C. Ends of the heater are covered with a thin layer of PG deposit

4 Temperature Uniformity at 1,500°C

A temperature uniformity investigation was conducted with the aim of checking the details of the furnace arrangement that might change or improve the temperature distribution at the temperature of the fixed point, such as the Re–C point (i.e., at about 2,500°C), and to establish the procedure to obtain a temperature gradient as small as possible at the cell position.

Because of difficulties in making temperature distribution measurements at high temperatures, the uniformity was investigated first at 1,500°C using two Type-R thermocouples. One of them was fixed in the rear channel and used for furnace temperature control and the other was moved through the front opening to scan along the furnace axis.

For the first stage, the arrangement of the furnace was as follows. The heater had the rings arranged in their original order, but they were PG-coated after vacuum operation (described in the preceding section). The cell holder was made of graphite, 80 mm in length, and wrapped with 3-mm graphite felt as an additional thermal screen. Inside the holder there were eight thin-sheet PG baffles, four in front of and four behind the cell.

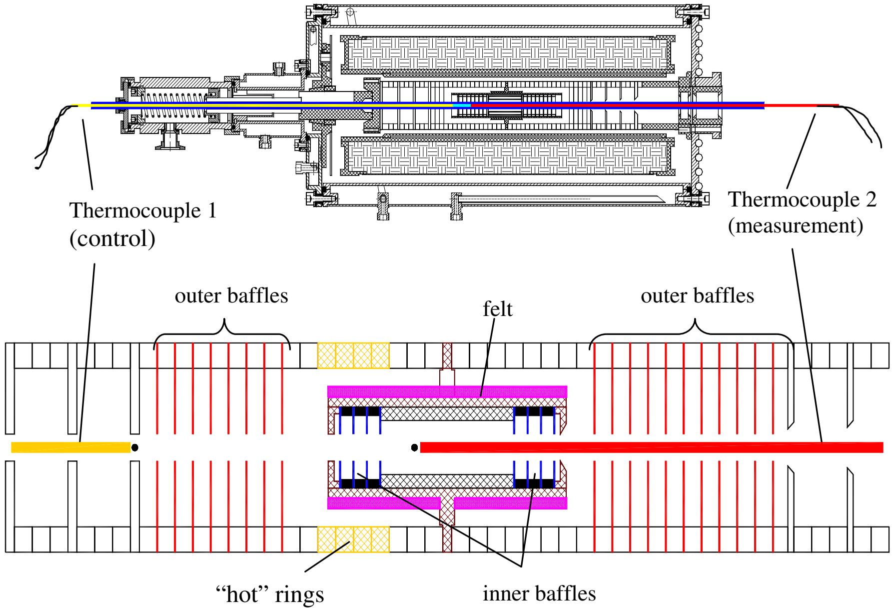

Fig. 5 Schematic of the measurement of temperature distribution at 1,500°C using Type-R thermocouples

No fixed-point cell was installed for any of the temperature uniformity measurements. In front of the holder, eleven thin-sheet PG material baffles were placed between the heater rings, and eight of these baffles were set up behind the holder. Figure 5 shows this arrangement.

Figure 6 shows the measured temperature distributions under various conditions. Curve 1 is the result of the measurement when all baffles and the felt wrapping were in place as described above. Then, the outer thin-sheet PG baffles were removed, retaining just the 4-mm thick baffles, and the distribution represented by curve 2 was obtained. Next, the outer baffles were put back, but the inner ones inside the holder were removed and curve 3 was measured. Curve 4 represents the situation when all the baffles were in place, but the felt wrapping was removed. One can see that the baffles (inner or outer) and the felt wrapping minimally influence the temperature distribution inside the cell holder.

Next, four rings around the rear end of the holder were replaced by ones with about twice the resistance (indicated in Fig. 3 as "hot rings"). The result (Curve 5) showed a significant change in the temperature distribution of about 40°C. This indicates that we can tune the furnace uniformity by placing different resistance rings around the cell position. Curve 6 was measured under the same conditions but without the graphite cell holder. By comparing curves 5 and 6, we can see that the graphite cell holder does not improve the uniformity.

In the second stage, we used a cell holder made of a 3 mm thick C/C tube, 150 mm in length, wrapped with 4 layers of graphite cloth. The measured temperature distributions are shown in Fig. 7. The first distribution had the high-resistance rings at the rear end of the cell position, but without the holder (curve 2; similar condition to curve 6

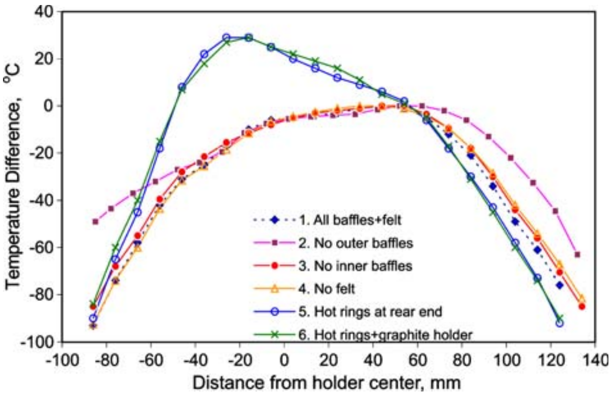

Fig. 6 Temperature distributions measured by a thermocouple at 1,500°C for the BB3500YY furnace with a graphite holder installed

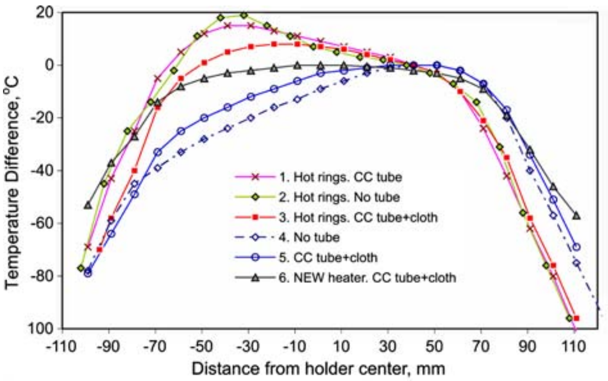

Fig. 7 Temperature distributions measured by a thermocouple at 1,500°C for the BB3500YY furnace with a C/C-tube holder installed

in Fig. 6). Then, we tried the same arrangement, but with the C/C tube in place, first without the cloth wrapping (curve 1), and then with the cloth wrapping (curve 3). A comparison of the curves shows that, in contrast to the graphite cell holder, the C/C-tube cell holder can improve the temperature uniformity. However, the cloth wrapping has a more significant effect than the C/C tube itself. Curves 4 and 5 represent distributions measured without and with the C/C-tube cell holder, respectively, but with low-resistance rings around the rear end of the cell position.

Finally, a new set of rings with the original ring-resistance distribution shown in Fig. 3 replaced the heater that had suffered from operation under vacuum. The new heater in combination with the C/C holder demonstrated much better uniformity than the previous one. By introducing some "hot" rings around the cell position, it was possible to achieve temperature uniformity within 5°C over a distance of 110 mm (curve 6 in Fig. 7).

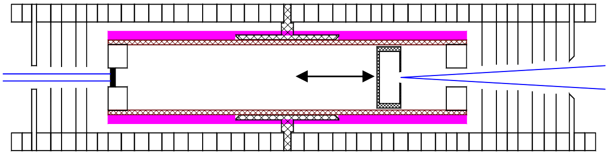

Fig. 8 Schematic of the measurement of the temperature distribution at 2,500°C using two radiation thermometers

5 Temperature Uniformity at 2,500°C

Two radiation thermometers were used for the temperature distribution measurements at higher temperatures. A schematic of the setup is shown in Fig. 8. One of them, a narrow-beam optical-fiber radiation thermometer that looks through the rear channel of the furnace to a blind baffle placed at the rear end of the C/C-tube cell holder, is used for furnace temperature control. The second thermometer looked through the front opening and was sighted on a movable target inside the holder. The target was a 10 mm length graphite tube with a soot-blackened bottom at one end and a 5 mm aperture at the other.

First, the target was placed inside the C/C tube near its front end, the furnace was brought to the desired temperature of 2,500°C, and the temperature of the target was measured by means of the second radiation thermometer. Then, the furnace temperature was set to 1,500°C, the target was pushed using an alumina rod and moved by 10–20 mm, the furnace was heated again to 2,500°C, and the second point of the temperature distribution measurement was taken. This was repeated to cover the length of the C/C tube.

As a test of the validity of this method, the temperature distribution was measured at 1,500°C with the same furnace arrangement using both measurement methods, the thermocouple and the radiation thermometer, and the results were compared. Both methods agreed within 1°C (Fig. 9). When the radiation thermometer method was applied to measure the temperature distribution at 2,500°C, the measurements showed that the hottest point was in the center of the C/C tube and that the temperature gradually decreased towards its ends. The central 40 and 80 mm parts of the tube were uniform to within 2 and 5°C, respectively. These results were measured with the ring resistance order shown in Fig. 10.

6 Work with HTFP Cells

6.1 Re–C Plateau Observation

A Re–C cell of 45 mm length, 24 mm outer diameter, and 3 mm aperture cavity was heated in the BB3500YY furnace to observe melting and freezing plateaux with a radiation thermometer. The Re material was of 99.999% nominal purity (supplier: Johnson Matthey). The crucible was internally insulated with C/C sheet [7]. The cell contained 73.5 g of eutectic alloy. The same cell was then used to observe the plateaux in a VR10-A23 type-Nagano furnace, which has an operational temperature limited to

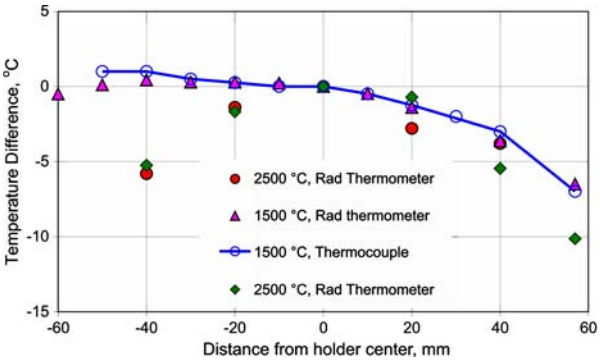

Fig. 9 Temperature distributions measured using the two radiation thermometer method for the BB3500YY with the optimized heater arrangement and C/C-tube holder

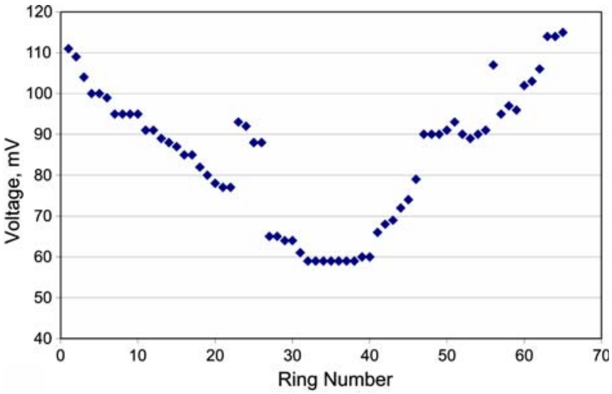

Fig. 10 BB3500YY resistance profile after uniformity improvement (Resistance is presented as the measured voltage drop across a 1.8 mm length of heater with 20 A current at room temperature). First ring is nearest to the front flange

2,500°C. Two LP3 radiation thermometers (S/N 80-38 and 80-40) were used throughout the investigation described in this paper, neither of which was calibrated precisely. Therefore, the temperature values of all plateaux presented in this paper have large (a few degrees) uncertainty.

The cell showed better melting-plateau shapes for a wider range of melting rates when installed in the BB3500YY furnace. For instance, using the heating/cooling furnace temperature settings of ±5 degrees relative to the fixed-point temperature, the BB3500YY could realize a plateau of 40 min duration. With a setting of ±10 degrees, a melting range of about 0.1 K was observed. Here, and later in this paper, the melting range was determined visually as the difference between the approximate start and end of a quasi-linear part of the melting curve (no mathematical algorithm was applied). Examples of the plateaux are shown in Fig. 11. With the VR10-A23, it was impossible to observe a similar quality plateau with furnace temperature settings smaller than ±10 degrees at this temperature because of its inferior temperature uniformity.

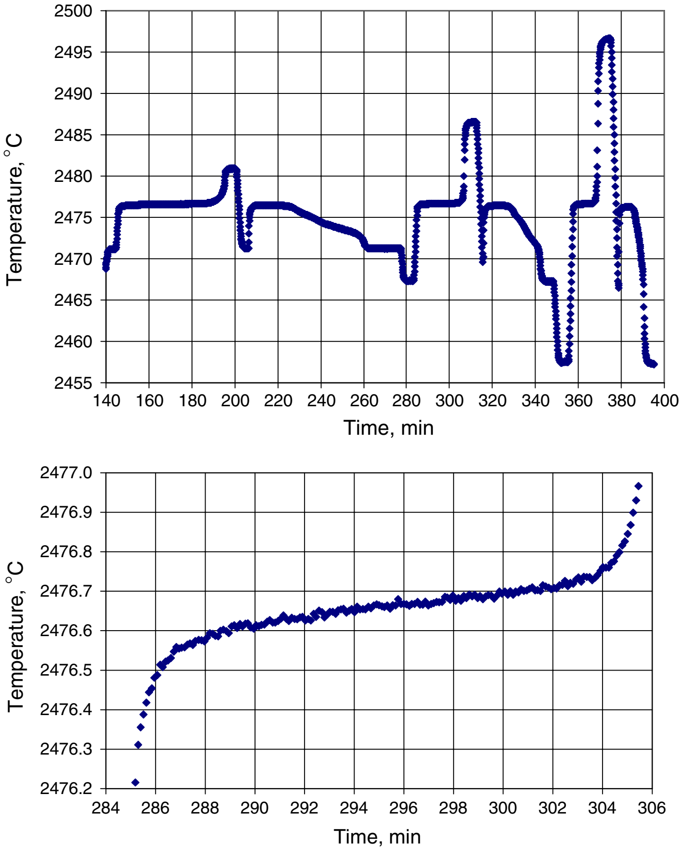

Fig. 11 Re–C cell plateaux observed in the BB3500YY using radiation thermometer LP3 S/N 80-38

6.2 Filling of Fixed-Point Cells

The BB3500YY was used to fill two crucibles: one with Re–C and another with TiC–C eutectic. For this, the furnace was operated vertically, with a cell holder specially designed so that it can be inserted and removed from the furnace without removing and disassembling the heater. A hole in the top part of the holder allowed the temperature of the fixed-point material to be measured with a radiation thermometer during the filling. The filling setup and the holder construction are shown in Fig. 12.

The Re–C cell made with the BB3500YY was then compared with two fixed-point cells filled in the Nagano furnace, all three filled with material from the same supplier (Johnson Matthey) and the same production lot of 99.999% nominal purity. The crucibles were identical in design, had an internal insulation of C/C sheet, and contained nearly the same amount of eutectic material: 62.4 g for cell ID 6SS-5 (filled in the BB3500YY), and 61.4 and 73.5 g for cells 6SS-2 and -4 (filled in the Nagano furnace). Cells 6SS-2 and 5 contained two layers of C/C sheet, while cell 6SS-4 contained one. The same furnace (BB3500YY) and the same radiation thermometer (LP3 serial

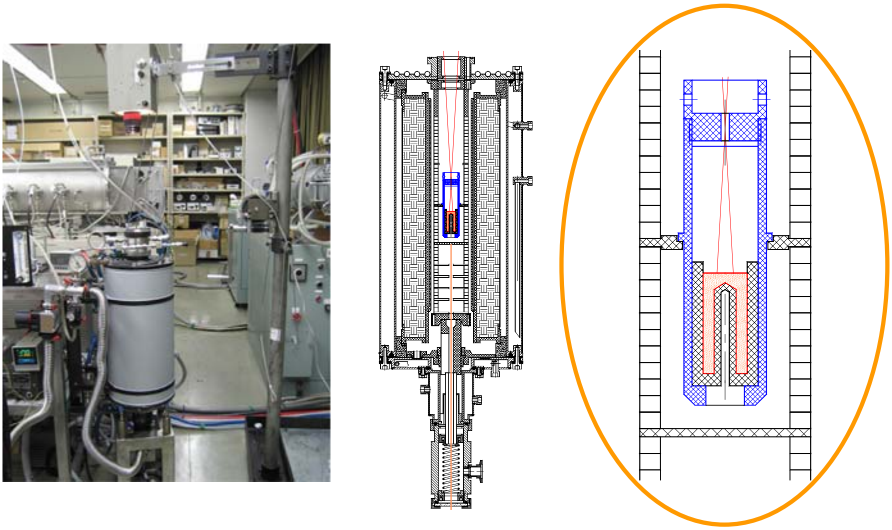

Fig. 12 HTFP cell filling setup

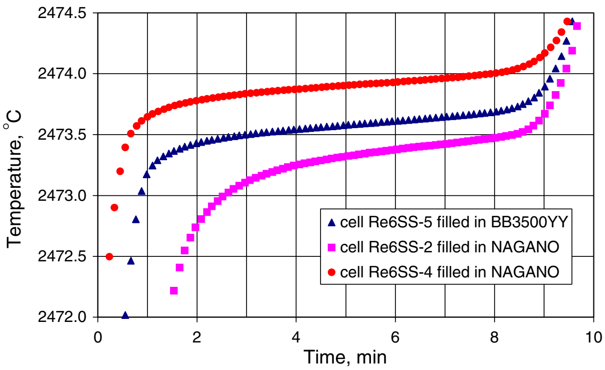

Fig. 13 Melting plateaux of Re–C cells made with the BB3500YY and NAGANO furnaces and measured using radiation thermometer LP3 S/N 80-40. Temperature values cannot be compared because of poor reproducibility of the measurements due to vignetting problems

number 80-40) were used for this comparison. Melting plateaux of the cells are shown in Fig. 13. Repeatability of the temperature measurements during this comparison was poor due to some vignetting of the radiation thermometer field of view. Therefore, only the shapes of the plateaux shown in Fig. 13 can be compared, not the temperature values. The plateau shape of the cell filled in the BB3500YY was no worse than those for the cells made in the Nagano, which indicates that no contamination occurred during the filling in the BB3500YY.

The TiC–C cell was compared with that made at VNIIOFI with a BB3200pg furnace. For both cells, the same source of Ti powder of 99.99% nominal purity was used. No C/C internal insulation was applied. Figure 14 shows a family of melting plateaux observed with an LP3 radiation thermometer. The shape of the TiC–C plateaux was

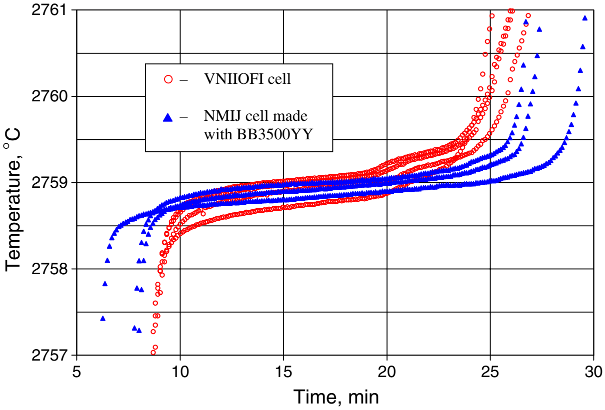

Fig. 14 Melting plateaux of TiC–C cells made with the BB3500YY and BB3200pg furnaces and measured using radiation thermometer LP3 S/N 80-40

generally poorer than for Re–C; nevertheless, with a melting range of about 0.3 K, these were the best TiC–C plateaux that have so far been observed by the authors. Although the cell made with the BB3500YY had slightly longer and flatter plateaux, both cells demonstrated similar behavior and the same melting temperature (within the melting-plateau repeatability and thermometer stability). Both cells seemed to show a slight degradation from melt to melt, which could not be explained and should be investigated in the future.

7 Conclusions

The investigations presented in this paper demonstrate that the temperature uniformity of a pyrographite furnace strongly depends on the PG heater rings' resistance and that the uniformity can be optimized for HTFP application by arranging the rings so that the resistance is lowest in the center of the heater and increases towards the ends and, additionally, by placing around the edges of the cell position some rings with resistance much higher than the adjacent rings. Thermal screens of graphite cloth wrapped around the cell holder can further improve the uniformity. Although the authors did not find any significant influence of the baffles on the temperature gradient, we believe that they are also useful.

Because of difficulties encountered in making temperature distribution measurements at higher temperatures, the furnace can be tuned at 1,500°C, where the temperature uniformity can be measured by platinum–rhodium-based thermocouples, as a good approximation. The method utilizing two pyrometers in combination with a moving target can give a more reliable temperature distribution measurement at higher temperatures.

The best uniformity the authors could achieve with the BB3500YY furnace, by applying the recommendations listed above, was 2°C over the 45-mm length of the Re–C cell. Tuned in such a way, the BB3500YY was able to realize the best Re–C and

TiC–C plateaux that have been observed so far by the authors; for instance, a Re–C melting plateau of 40 min duration and a melting temperature range of less than 0.2 K was obtained.

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