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Multizone Furnace for Analysis of Fixed-Point Realizations in the Range from 1,000°C to 1,700°C

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Abstract In this article, the development of a laboratory furnace specially designed for analysis of fixed-point plateau realizations in the range from 1,000 °C to 1,700 °C that enables control of various temperature distribution settings along the heating zone length is presented. A total of 13 thermocouples are built into the furnace tube wall to control the temperature as well as to measure the temperature distribution. The furnace is divided into seven independently controlled heating zones. Each heating zone comprises a $MoSi_2$ heating element and its dedicated DC power supply module. The furnace temperature is controlled by manipulating the output voltage of each power supply to control the temperature of each heating element, as estimated from its electrical resistance. The heating power and temperature measurement are fully controlled by a computer using an application written in Lab VIEW, allowing very flexible furnace control. The furnace can be used in air as well as in an inert atmosphere. Measurements of the temperature distribution of the furnace during a melting-point realization are presented.

Keywords Fixed-point cell · High temperature · Laboratory furnace · Temperature distribution

1 Introduction

Laboratory tube furnaces used for fixed-point realizations are normally optimized for a homogeneous temperature distribution within the heating zone. Since it is difficult for a single-zone furnace to guarantee good temperature uniformity, additional heating zones are typically added at each end of the furnace [1]. Adding heating zones

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with separate control results in a three-zone furnace where the end zones are used to compensate the heat losses from the center zone to each end of the tube. When the effect of thermal gradients in the furnace on the fixed-point realization needs to be investigated, a three-zone furnace is the minimum requirement.

Some authors have used their existing furnaces to purposely create non-uniform temperature distributions along the working tube of the furnace [2,3]. However, when existing laboratory furnaces are used for temperature non-uniformity analysis, their performance is limited either by the low number of zones or by the lack of proper control. A three-zone furnace enables the user to set the temperature at three points along the working tube, where the control temperature sensors are mounted. The low number of heating zones and control sensors allows the temperature to be controlled at only a small number of points, and therefore presents only limited control possibilities. A furnace with more than three independently controlled heating zones would be of benefit for the analysis of the effect of different temperature distributions on fixed-point realizations.

One of the most important requirements for good furnace control is the temperature sensor. Temperature sensors to control furnaces at high temperatures are usually thermocouples, although some furnaces also use fiber-optic radiation thermometers. In any case, the temperature distribution in the working tube greatly depends on the performance of the control thermometer. Control thermometers should provide good stability, and they should measure the temperature as close to the working tube as possible. While fiber-optic radiation thermometers directly measure the temperature of the furnace tube, they are not as simple and robust as a thermocouple. However, one of the problems in using thermocouples for temperature control is their position within the furnace, which is somewhere between the furnace tube and the heating elements. This usually results in a temperature difference between the thermocouple and the inside of the furnace tube. Even if the control thermocouples read the same temperature, the actual temperature distribution in the working tube deviates from the temperature maintained by the controller.

One way of determining the actual temperature distribution is by using two thermocouples inserted into the working tube of the furnace. One thermocouple is kept at a fixed location to measure the temperature drift of the furnace. The other thermocouple is movable and is used to measure the temperature at a number of locations within the furnace.

In order to use this procedure, the fixed-point cell is usually removed from the furnace, since there is generally not enough space in the furnace tube to accommodate the cell and the two thermocouples. As a result, the temperature distribution measurements are performed either with an empty working tube or with a block with axial holes for the thermocouples inserted into the furnace tube. Either way, the temperature distribution during the fixed-point realization is not monitored and can only be assumed to be identical to, or at least similar to, the one previously measured.

2 Furnace Construction

We have constructed a special furnace where we combined the added flexibility and control of the temperature distribution provided by a greater number of zones with the

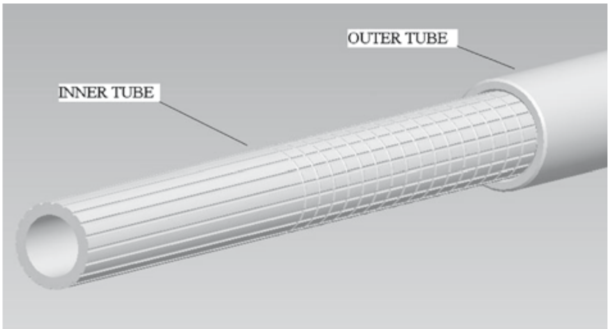

Fig. 1 Inner and outer tube assembly

ability to continuously measure and display the temperature distribution [4]. Additional heating zones enable active control of the temperature distribution, even for a short heated length, whereas a three-zone furnace relies on its geometry and longer heating zones. The furnace can be used for fixed-point realizations in air as well as in an inert atmosphere.

The core of the furnace comprises insulation, the furnace tube, and heating elements. The length of the furnace core is small to prevent bending of the furnace tube at high temperatures under its own weight. The furnace is divided into seven independently controlled heating zones. Each heating zone comprises a heating element, a dedicated DC power supply module, and a control thermocouple. Three layers of different refractory ceramic-fiber boards were used for insulation, each with a thickness of about 25 mm. The maximum operating temperatures of the employed insulation boards range from 1,200 °C to 1,700 °C. The material chosen for the furnace tube was 99.7% pure alumina. The furnace tube is constructed from two parts, an inner tube and an outer tube. The inner tube has axial and radial grooves machined into its outer surface as shown in Fig. 1. The grooves serve to support and guide the thermocouple wire. Thermocouples made from Type-R thermocouple wire were positioned in the grooves. The hot junctions of the thermocouples are positioned in a straight line along the bottom part of the furnace tube with a spacing of 25 mm between them. The 13 measuring points cover a length of 300 mm. When assembled, the tubes form a furnace tube with an inner diameter of 25 mm, an outer diameter of 45 mm, and a length of 700 mm. Thermocouple wires exit the furnace tube at each end, where they are connected to compensating extension leads.

Standard U-shaped $MoSi_2$ heating elements were chosen for their high maximum operating temperatures of over 1,800 °C in an oxidizing environment. Each heating element is suspended vertically from the furnace top insulation and placed in such a way that the lower part of the U-shape encircles the furnace tube. The spacing between each heating element is 50 mm so that the heated length of the tube is about 350 mm. The elements have a very low electrical resistance and require low supply voltages and high currents.

Each heating element is connected to a commercially available single-output, 1500 W switched-mode DC power-supply unit. Each power-supply unit has a variable output voltage of up to 12 V and a rated output current of up to 125 A. The power supplies feature remote output control, overload and over-temperature protection, and power factor correction. The power supplies are mounted at the bottom part of the housing. The width of the furnace is about 40 cm, the length is about 70 cm, and the height is about 100 cm.

3 Control System

The control system consists of a personal computer with LabVIEW control software, various I/O boards, and seven independent control subsystems. Each subsystem comprises a DC power supply, a shunt resistor, and a heating element. The system parts that were chosen offer great flexibility and facilitate future upgrades. The furnace operates without dedicated control thermocouples and without commercial controllers.

There are three measured parameters that are used for the control of each heating zone: the voltage drop across the heating element U_e, the voltage drop across the current shunt resistor U_s, and the output of the corresponding thermocouple in the wall of the furnace tube. The current I_e for each heating element is calculated from the voltage U_s and the known shunt resistance. The power and the electrical resistance for each heating element are calculated from the voltage U_e and the current I_e. The power of the heating elements is controlled by adjusting the output voltage of the power supplies. It is necessary to know the supply current in order to prevent the output of the power supply from reaching its maximum current limit. During the start-up of the furnace, the low electrical resistance of the heating elements requires large currents that are well above the power-supply specifications. The calculated power is used to limit the power supplied to the heating elements in order to reduce the maximum load and prolong their lifetime.

The electrical resistance of a $MoSi_2$ heating element depends strongly on the element temperature [5]. The resistance curve increases sharply with temperature, and this can be very useful in estimating the heating element temperature from its calculated electric resistance. The temperature of the heating element was measured by an optical pyrometer. A second-order polynomial was fitted to the recorded curve, and it serves as the function used to estimate the heater temperature from its calculated resistance.

A simple closed-loop control algorithm was used to control the element temperature to a value based on its estimated temperature. In this way, stable control of the furnace is possible without any dedicated temperature sensors. However, a backup thermocouple should monitor the furnace temperature to prevent overheating of the furnace in case of failure of the control system. In Fig. 2, the temperature distribution measured by thermocouples in the furnace tube is shown with all heating elements set to the same value (1,100 °C, 1,300 °C, and 1,500 °C). The temperature distribution in this case is similar to the performance of a single-zone furnace. The temperature inside the furnace as measured by a thermocouple was found to be within 1 K over a period of 1 h once the furnace reaches its operating temperature. During nine months of furnace operation, we observed a drift of the temperature in the furnace of about 10 K with the temperature control based on the calculated heater temperature.

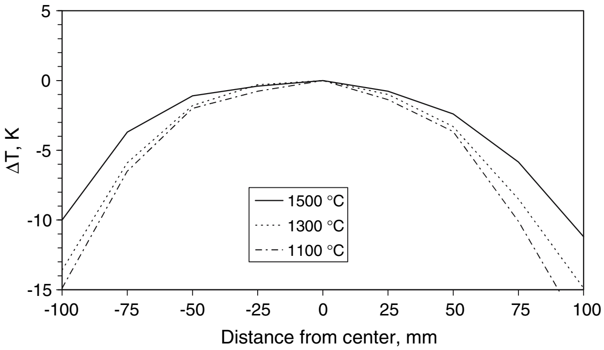

Fig. 2 Temperature distribution in the furnace with heater-temperature control

For fixed-point realizations, the heater temperature control can be used in conjunction with a thermocouple or pyrometer measuring the temperature inside the cell. The data from the thermocouple or pyrometer serve as absolute temperature information, and the furnace can be controlled by adjusting the heating element temperature relative to the measured temperature.

The temperature distribution during a fixed-point realization is measured by 13 thermocouples built into the wall of the working tube of the furnace. Since the number of thermocouples is greater than the number of zones, any of the 13 thermocouples can be used for control, but thermocouples positioned in the center of each heating zone are normally selected. The other six thermocouples are positioned between two neighboring zones and are used only for measurement.

To control the temperature distribution in the furnace tube, a secondary PID control loop is added. The secondary controller controls the temperature at the location of chosen thermocouples in the wall of the furnace tube by modifying the set point for the heating-element temperature. The performance of the furnace with the added control loop is shown in Fig. 3 for a uniform temperature distribution and four different temperature gradients at 1,500 °C. The effective length over which the uniform axial temperature distribution remains within 0.2 K is about 150 mm. The radial uniformity inside the tube as measured by a thermocouple is within 0.2 K and remains stable to within 0.3 K for a period of 1 h.

The furnace can be controlled in either a closed-loop or an open-loop mode. During closed-loop control, the temperature measured by thermocouples corresponding to each zone is controlled to generate the required temperature distribution. The open-loop control mode can be used during fixed-point plateau realization. When the furnace temperature is controlled by directly setting the heating-element temperature, all of the thermocouples in the wall of the tube can be employed to measure the actual temperature along the fixed-point cell.

4 Results

Testing of the furnace was divided into three phases. In the first phase, the heating performance was tested up to the maximum temperature. Throughout the initial testing, a normal furnace tube without grooves was used. Testing included heating the

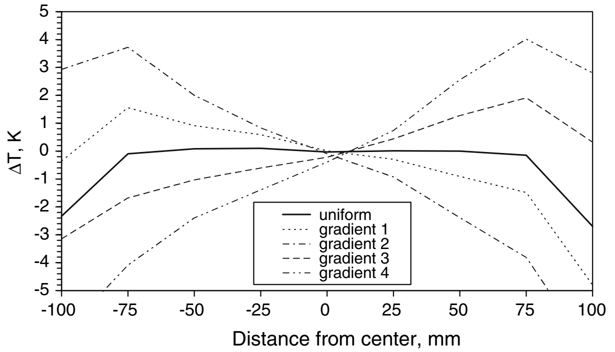

Fig. 3 Uniform temperature distribution and temperature gradient generation at 1,500 °C

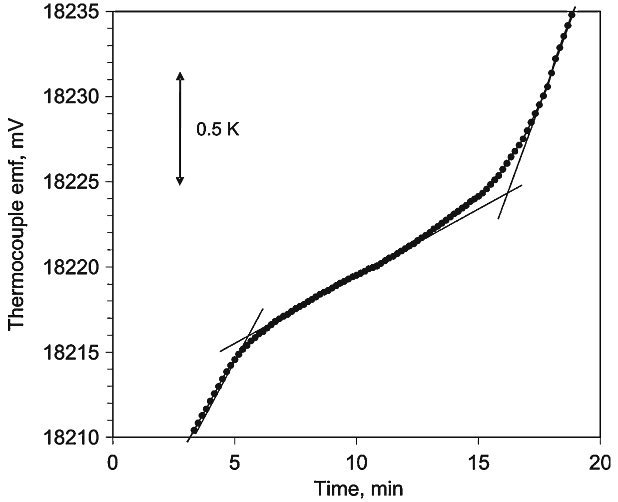

Fig. 4 Melting plateau of a palladium miniature fixed-point cell

furnace to above 1,600 °C for about 2 h. In the second phase, the melting point of a miniature palladium fixed-point cell was realized inside the furnace in an air atmosphere several times, using only manual set-point control of the heater temperature, as described in the previous section. A Type-R thermocouple, previously calibrated by the wire-bridge method (calibration performed by NMi Delft Netherlands in 1999) with an uncertainty of 1.5 K, was used to detect the melting point of the palladium cell. The result of the melting-plateau realization is shown in Fig. 4. The melting-point temperature was obtained from the beginning of the melting plateau [6]. The difference between the previous calibration of the thermocouple and the measurements in our new furnace at the melting point of palladium is 2 μV, which is equivalent to about 0.2 K. The estimated uncertainty of the realization in the developed furnace is 1.5 K [7].

In the third phase, the normal furnace tube was replaced with the special furnace tube (Fig. 1) with built-in thermocouples in order to test the temperature distribution measurements. To evaluate the temperature distribution measurement capability during a fixed-point realization, we used an experimental fixed-point cell built in our laboratory. The cell is intended to be used in air, as a reference in an industrial environment, but is still under development and has not yet been fully investigated. It is based on copper oxidized in air in an alumina crucible. During oxidation, a eutectic is formed with a reproducible melting-point temperature of about 1,066 °C. The cell has a length of 80 mm and a diameter of 23 mm. The cell was positioned in the center of the heating zone above thermocouples 6, 7, and 8. A Type-R thermocouple was inserted into the cell. The furnace was controlled only by heater temperature control so that all of the built-in thermocouples could be used for the temperature distribution measurement. All heating elements were set to the same temperature. The furnace temperature was stabilized about 8 K below the melting point and then raised to about 8 K above the melting-point temperature to realize the plateau. This temperature difference is rather high, but it emphasizes the effect of thermal gradients at the expense of a shorter melting plateau.

Figure 5 shows the melting plateau and measurements of thermocouples number 6, 7, and 8 (TC6, TC7, and TC8, respectively). The thermocouple positions with respect to the cell are also shown at the bottom. As the thermocouples are positioned close to the cell, they detect the change of phase of the material in the cell. The thermocouple output was corrected to align the curves at the plateau. The deviation between thermocouples 6, 7, and 8 before and after the melt is about 1 K. At the beginning of the melt, the plateau is relatively flat but there is a break-off point in the middle of the plateau. The point corresponds to a temperature rise of thermocouples 7 and 8. Only when the material is totally melted the rise of thermocouple 6 can be observed. This suggests that the melting finished first at the position of thermocouple number 8, immediately after

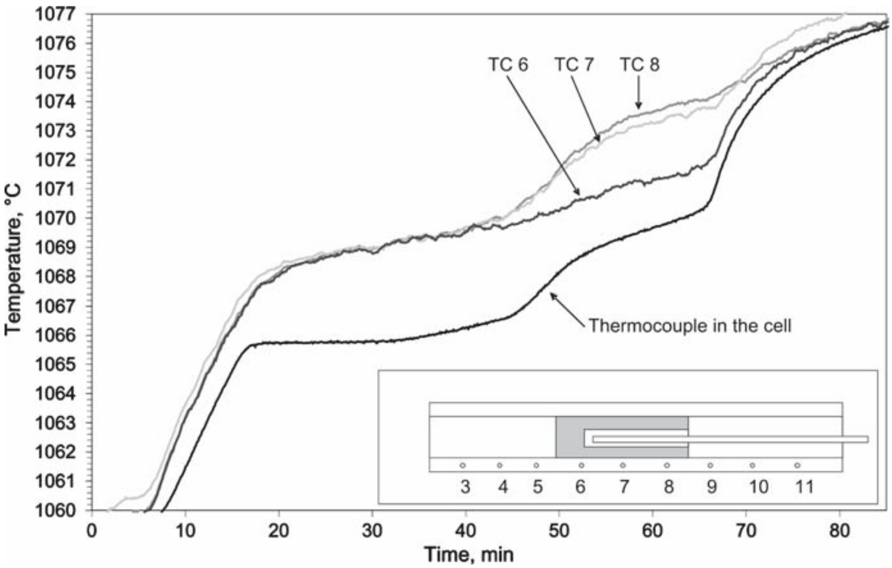

Fig. 5 Realization of the melting plateau

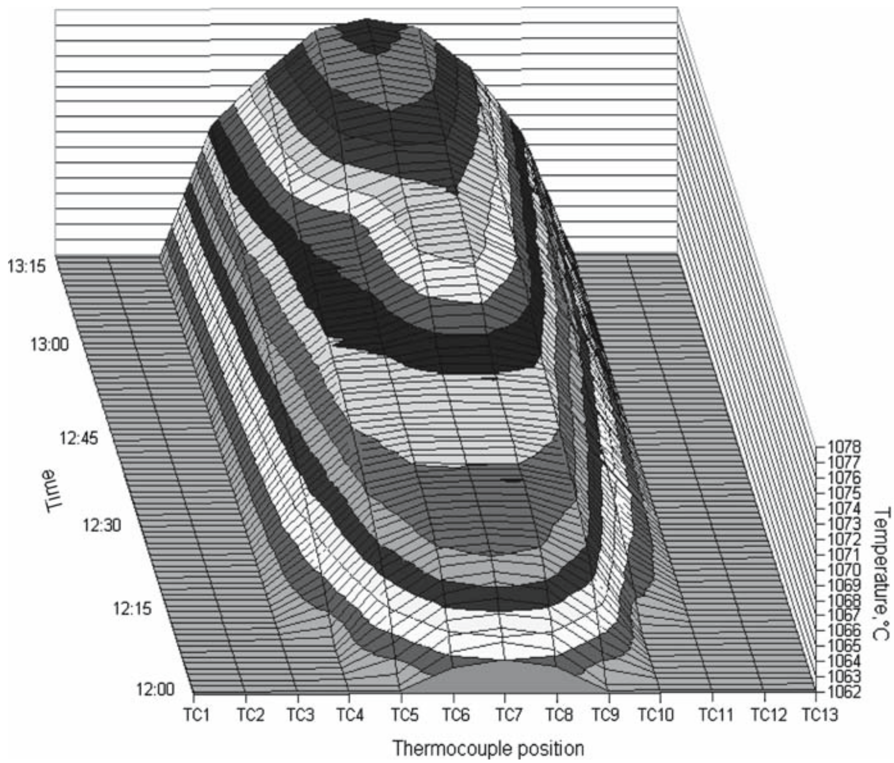

Fig. 6 Temperature distribution in the furnace tube during melting

that at thermocouple number 7, and lastly at position 6. The break-off point should correspond to the thermal bridge created between the inner and outer cell walls when the material has locally melted. The output of the thermocouple inserted into the cell rises first with thermocouples 7 and 8 but remains influenced by melting of material at the position of thermocouple 6. When the material finishes melting at the position of thermocouple 6, all thermocouples show a temperature increase.

The temperature distribution measured by all thermocouples in the furnace tube during the melting-point realization is shown in Fig. 6. It can be seen that there is a temperature gradient in the furnace tube, but the temperature increases quite uniformly. The thermocouples detect the plateau as the rate of temperature increase slows, but thermocouple 6 remains influenced by the melting metal even while the temperature at positions 7 and 8 starts to rise in the middle of the plateau. The deviation of all three thermocouples equalizes again when the material has entirely melted.

5 Conclusion

The development of a seven-zone furnace for the analysis of fixed-point realizations was presented. Thermocouples built into the furnace tube wall showed good performance during control of the temperature distribution in the furnace as well as during measurement of the actual temperature distribution. With the presented system, real-time measurements of thermal gradients in close proximity to the fixed-point cell are possible, even during realizations of a melting or freezing plateau. An example of such a measurement during a melting plateau was given, and the results showed that it is possible to detect thermal gradients along the fixed-point cell during the realization. The measured temperature distribution along the fixed-point cell gives insight into the dynamics of the phase change transition inside the cell. The system offers great potential for the analysis of the effect of thermal gradients during fixed-point realizations. Future investigations will be done to analyze the effect of thermal inhomogeneity on the fixed-point plateau. Measurements with different temperature profiles are planned and different cells will be used.

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References

1. *Techniques for Approximating the International Temperature Scale of 1990* (BIPM, 1990), pp. 16–18
2. Y. Yamada, N. Sasajima, H. Gomi, T. Sugai, in *Temperature: Its Measurement and Control in Science and Industry*, vol. 7, ed. by D.C. Ripple (AIP, New York, 2003), pp. 985–990
3. J. Fischer, H.J. Jung, R. Friedrich, in *Temperature: Its Measurement and Control in Science and Industry*, vol. 6, ed. by J.F. Schooley (AIP, New York, 1992), pp. 53–57
4. M. Hiti, *Development and Analysis of Multizone High-temperature Furnace for Realization of Temperature Fixed-points*, Ph.D. Thesis, University of Ljubljana, 2007
5. *Kanthal Super Handbook* (Kanthal AB, Sweden, 1999)
6. F. Edler, in *Proceedings of TEMPMEKO '96, 6th International Symposium on Temperature and Thermal Measurements in Industry and Science*, ed. by P. Marcarino (Levrotto and Bella, Torino, 1997), pp. 183–188
7. J. Bojkovski, V. Batagelj, M. Hiti, in *Proceedings of the Thirteenth International Electrotechnical and Computer Science Conference ERK 2004*, vol. A, ed. by B. Zajc (IEEE Region 8, 2004), pp. 443–446