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Design of a sample holder for spectral- and angle-resolved emissivity measurement under vacuum at temperatures up to 1000 °C

Max Reiniger, Albert Adibekyan, Christian Monte, Jörg Hollandt
Physikalisch-Technische Bundesanstalt, Abbestraße 2-12, 10587 Berlin
Max.Reiniger@ptb.de

Abstract

The facility for emissivity measurement under vacuum at the Physikalisch-Technische Bundesanstalt (PTB) is able to measure the directional spectral emissivity in the wavelength range from 4 µm to 100 µm and in the temperature range from -40 °C to 450 °C. Here we present the design of a new sample holder for the extension of the temperature range of this facility up to 1000 °C. We will discuss the concept of heating and the issues of getting a homogeneous temperature distribution on the sample surface. We describe the solutions which are necessary to obtain a homogeneous temperature distribution on the sample surface even at very high temperatures up to 1000 °C.

Key words: spectral emissivity, sample holder, under vacuum, temperature up to 1000 °C, homogeneous temperature distribution.

Introduction

The accurate knowledge of the emissivity of a material is a requirement for a wide range of thermophysical applications like determining its radiation temperature or calculating its radiation budget. The temperatures of interest vary from low temperatures found under space conditions up to high temperatures for industrial applications. Examples of applications which require the knowledge of emissivity with low uncertainty are e.g. absorbers for solar thermal electricity generation or process control by radiation thermometry in glass-, plastic- or metal processing. The Physikalisch-Technische Bundesanstalt (PTB), the national metrology institute of Germany, provides emissivity measurements over a wide range of temperatures. Currently the measurement facility under vacuum covers a temperature range from -40 °C to 450 °C and a wavelength range from 4 µm to 100 µm [1]. A dynamic emissivity measurement facility covers a temperature range from 800 °C to 2000 °C at a wavelength of 1064 nm [2]. To bridge the gap in temperature ranges and to enable the direct comparison of both facilities we designed and optimized a sample holder for the temperature range from 50 °C to 1000 °C.

Requirements

Essential for low uncertainties in emissivity measurements is a homogeneous temperature distribution on the sample surface. The main task of the development of the new high temperature sample holder was to find an optimized material, sample holder geometry and heating concept to reach the desired temperature of 1000 °C with the best possible temperature homogeneity on the sample surface. The sample itself, i.e. its surface emissivity, affects the temperature homogeneity. The challenge was to evolve a heating concept which is able to vary and optimize the surface temperature homogeneity for various surface emissivities.

Simulations

We used COMSOL Multiphysics V4.4 for the thermal simulations. The thermal conductivity and emissivity of the sample holder, given by the selected material, its geometry and heating concept were systematically varied to find an optimized solution. Simulations were done for three different materials: Inconel® alloy 600, Nickel 200 and pure molybdenum with different geometries and multiple heating zones. The geometry of the sample holder is limited by the existing facility. Accordingly, the maximum size of the sample holder is a cylinder with a height of about 40 mm and a diameter of about 70 mm. The typical sample is a cylinder with a height or thickness of 5 mm and a diameter of 50 mm. These dimensions were used for the iterative optimization of the sample holder.

Heating concept

The simulations started with one heating zone on the base of the cylinder. The result was, as expected, an inhomogeneous temperature distribution on the sample surface. So it was necessary to have multiple heating zones to adjust the homogeneity on the sample surface. The next step included a second heating zone on the cylinder shell. This additional heating zone enables us to better control the temperature homogeneity on the sample surface via different power ratios between the two zones.

Sample holder material

Other parameters considered were the thermal conductivity, melting point and surface emissivity of the sample holder material. The simulations demonstrated the importance of a sufficiently high thermal conductivity to reach the aimed temperature up to 1000 °C. Due to the maximum operating temperature of 1055 °C of the chosen heating wire it was necessary to keep the temperature difference between the heating wire and the sample surface small. As a result of our simulations we chose molybdenum which allows reaching the desired temperature of 1000 °C at the sample surface due to its higher thermal conductivity compared to INCONEL® 600 and Nickel 200. Its lower emissivity compared to the other two materials also reduces the required heating power for reaching 1000 °C.

Radiation shield

The undesirable heat loss of the sample holder and the high thermal radiation load on the spherical sample enclosure can be reduced by radiation shields. Simulations were done with different radiation shield materials featuring different surface emissivities to quantify their effect. It was found that two gold coated nested shields allow reaching sample temperatures of 1000 °C with an emissivity of the sample $\varepsilon = 0.8$ applying just 500 W of heating power.

Geometry improvement

The two heating zones allowed adjusting the temperature distribution on the sample surface but the distribution was still not sufficiently homogeneous and the adjustment range to compensate for varying sample emissivities and temperatures still too small. It was necessary to shape the “initial temperature distribution” by a geometrical improvement. This was achieved by a circular groove between the mounting surface and the two heating zones (Fig. 1 and Fig. 2). The size and position of this groove was optimized for a typical sample emissivity of $\varepsilon = 0.4$.

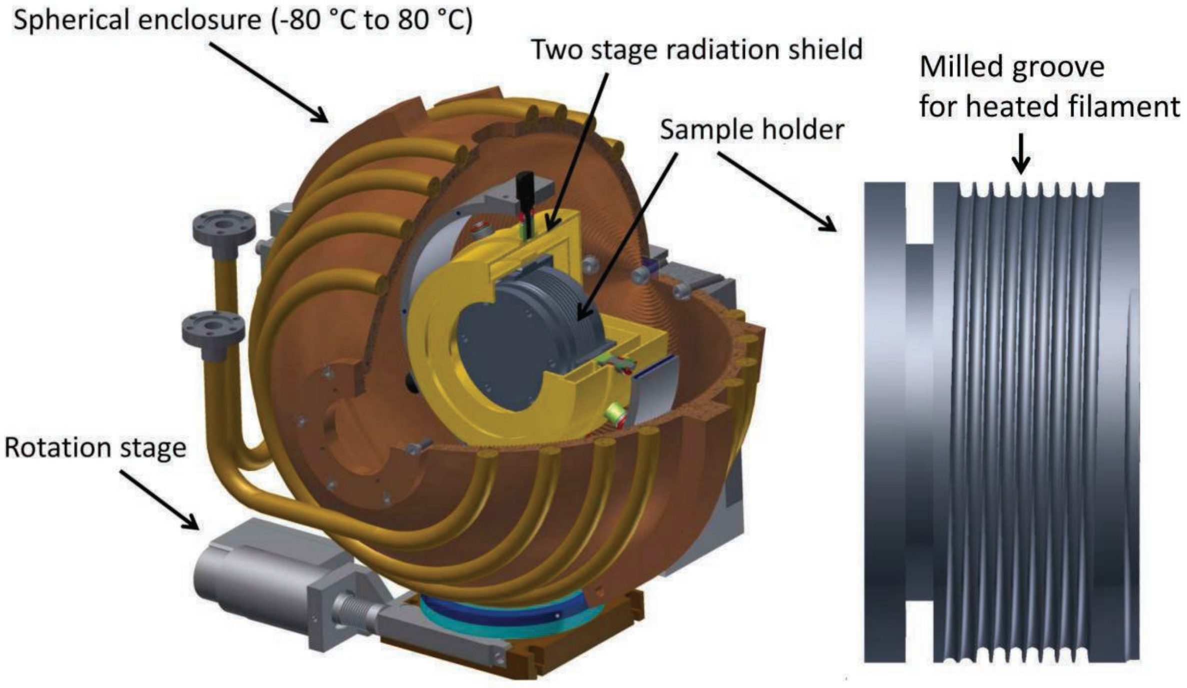

Fig. 1 *Sketch of the overall setup. The optimized sample holder and a two stage radiation shield are mounted in the existing enclosure. The sample holder features milled grooves for two bifilar coiled heating wires and a circular groove for optimized temperature homogeneity on the sample surface.*

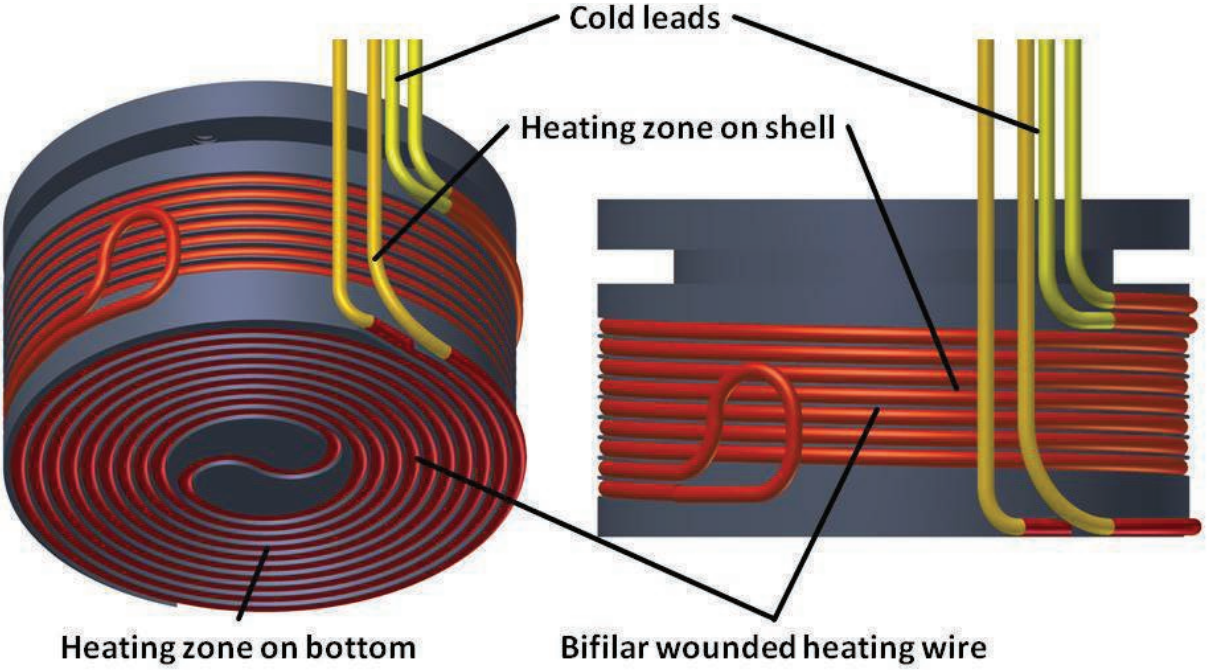

Fig. 2 *Sketch of the sample holder with heating wires. The new sample holder features milled grooves for two bifilar coiled (for reduction of the produced electromagnetic field) heating wires. The heating wires are soldered into the milled grooves for a better thermal conduction.*

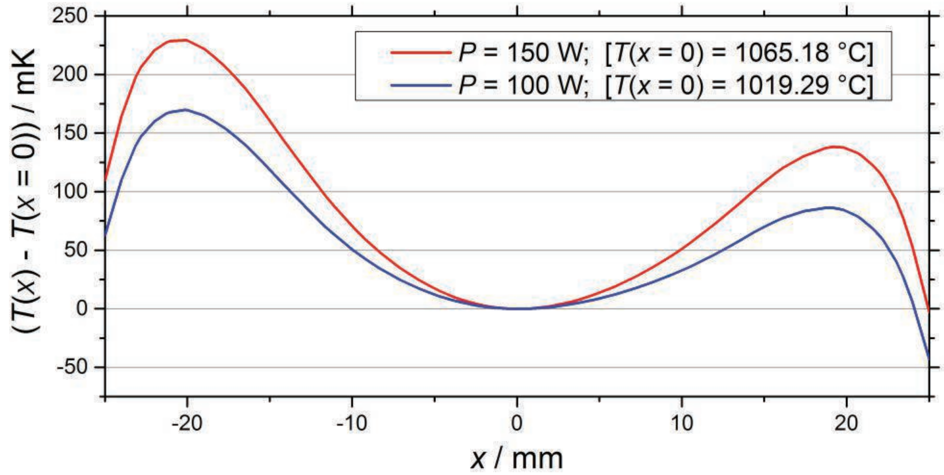

Fig. 3 *Calculated temperature distribution on the surface of a sample with an emissivity of ε = 0.4. Different heating powers are applied at the cylinder shell and a constant heating power of 250 W at the bottom. The unsymmetrical trend results from the asymmetry induced by the threefold (120°) mounting of the sample holder.*

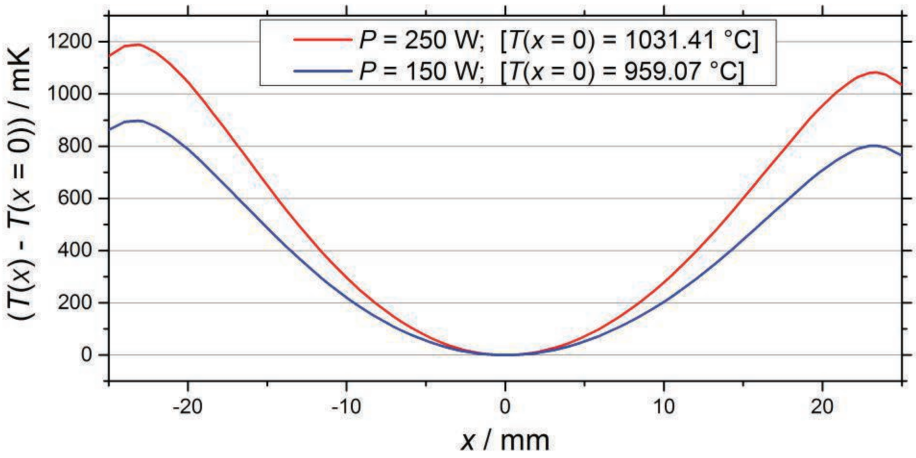

Fig. 4 *Calculated temperature distribution on the surface of a sample with an emissivity of ε = 0.8. Different heating powers are applied at the cylinder shell and a constant heating power of 250 W at the bottom. Again the unsymmetrical trend results from the asymmetry induced by the threefold (120°) mounting of the sample holder.*

Final design

The final setup consisting of the optimized sample holder with its two stage radiation shield inside the already existing enclosure is shown in Fig. 1. A more detailed sketch of the new heater of the sample holder is shown in Fig.2. The spherical enclosure shown in Fig. 1 is coated with Nextel-Velvet-Coating 811-21 with an emissivity of $\varepsilon = 0.97$ [3] and can be temperate controlled between -80 °C and 80 °C.

Results

Fig. 3 and Fig. 4 illustrate for a surface temperature of about 1000 °C surface temperature distributions of samples with an emissivity of $\varepsilon = 0.4$ and $\varepsilon = 0.8$. Shown are examples with different heating powers on the cylinder shell and a constant heating power of 250 W applied on the bottom of the holder. For a sample emissivity of $\varepsilon = 0.4$ (Fig. 3) a temperature homogeneity of about 200 mK is achieved. In this case a total power of 350 W is needed to reach the temperature of 1000 °C. In Fig. 4 the simulation results for a sample emissivity of $\varepsilon = 0.8$ are shown. Here the achieved temperature homogeneity is better than 1.2 K. The temperature of 1000 °C is reached with a total heating power of around 500 W.

Conclusion

The concept of the new sample holder features two heating zones one at the back side and one at the outer shell of a cylindrical molybdenum body. The optimized geometry has a circular groove between the mounting surface and the two heating zones as shown in Fig.1 and Fig. 2. The depth of the groove is optimized for the best temperature homogeneity of a sample with an emissivity of $\varepsilon = 0.4$. The sample holder is made from molybdenum which allows reaching the desired temperature of 1000 °C at the sample surface due to its high thermal conductivity and its low emissivity without exceeding the maximum operating temperature of 1055 °C for the heating wires. Additionally, we have designed an optimized two stage radiation shield to reduce radiation losses of the holder and by this to lower the necessary heating power for reaching a maximum temperature of 1000 °C. The temperature homogeneity on the sample surface at 1000 °C will be better than 1.2 K for all sample emissivities. The new sample holder closes the gap in temperature ranges of the spectral emissivity setups at PTB between 450 °C and 800 °C and, furthermore, allows comparing the static and the dynamic emissivity measurement approaches at PTB. The first experimental results will be present at the IRS² conference.

References

[1] A. Adibekyan et al., Emissivity measurement under vacuum from 4 µm to 100 µm and from -40 °C to 450 °C at PTB, *Int. J. Thermophysics*, 2014, 10.1007/s10765-014-1745-7

[2] S. Krenek et al., A Study on the Feasibility of Measuring the Emissivity with the Laser-Flash Method, Int. J. Thermophysics (2010) 31, 998-1010

[3] J. Lohrengel, R. Todtenhaupt, Wärmeleitfähigkeit, Gesamtemissionsgrade und spektrale Emissionsgrade der Beschichtung Nextel-Velvet-Coating 811-21 (RAL 900 15 tiefschwarz matt); *PTB-Mitteilungen 106 Heft 4 (1996)*; 259-265