



# Construction and preliminary testing of a guarded hot plate apparatus for thermal conductivity measurements at high temperatures



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## ABSTRACT

We constructed a guarded hot plate (GHP) apparatus that can be used for thermal conductivity measurements from 100 °C to 1000 °C. The apparatus measures the effective thermal conductivity of square-shaped thermal insulation materials of 300 mm per side in a controlled gas environment. As a preliminary test for the high temperature GHP apparatus, we first measured the amount of electrical power required for heaters at various locations to maintain the temperature at up to 900 °C. After this, two specimens of thermal insulation material whose thermal conductivities are  $\approx 0.044 \text{ W} \cdot \text{m}^{-1} \text{ K}^{-1}$  and  $\approx 0.025 \text{ W} \cdot \text{m}^{-1} \text{ K}^{-1}$  at room temperature were tested at a thermally steady state with unilateral heat flow at temperatures between 100 °C and 800 °C. We applied an offset to the guard plate to actively compensate for the lateral heat flow across the air gap. The measurements showed that the linearity between the temperature difference on the two sides of the specimen and the power dissipated across the specimen was extremely good (with a standard deviation of 0.1%). After some modifications suggested from the preliminary test, we expect that the high temperature GHP will work within the technical specification CEN/TS 15548-1:2014 in the range up to 800 °C, but rigorous uncertainty assessment is yet to be performed after such modifications.

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## 1. Introduction

Thermal conductivity is an important property of materials used in housing, industries, and commercial buildings. Among several methods used to measure the thermal conductivity accurately, the guarded hot plate (GHP) method is a primary way for low-to-medium thermal conductivity materials because it is an absolute measurement method. This means that the traceability of the GHP measurement comes from the traceable measurements of length, temperature, and electrical power (current and voltage) [1]. The physical principles behind thermal conductivity measurements with a GHP are well known, and there are international standards already in place to guide the implementation of these [2–4]. Furthermore, the standards lay out guidelines for the specification, design considerations, operating parameters, and expected performance for the thermal conductivity measurements of various specimens.

Fig. 1 summarizes the physical principles of the GHP method in a double-sided heat flow configuration. In this configuration, the geometry of the sample specimen, whose thermal conductivity is

to be measured, and the plates are placed in a symmetric arrangement with respect to the horizontal plane at the elevation of the vertical center of the hot plate (HP). For the two sheets of specimen located above and below the HP, the temperature on the hot side is set to  $T_h$  by controlling it at the HP and the guard plate (GP), and on the cold side to  $T_c$  ( $< T_h$ ) by controlling it at the cold plates (upper cold plate, UCP; and lower cold plate, LCP). The term ‘cold’ plate indicates that the temperature is lower than the average temperature of the specimen, which is approximately  $T_m = (T_h + T_c)/2$ . In this configuration, there is one-dimensional heat flow from the HP and GP to the two cold plates, represented by thick vertical arrows pointing away from the HP and GP in Fig. 1. The auxiliary guard heaters are controlled to  $T_m$  to minimize heat loss near the edge of the sample and to keep the heat flow as close to the one-dimensional direction as possible. Any deviation from the one dimensionality of the heat flow results in error in the thermal conductivity measurement.

In the present paper, we denote “heater plates” to collectively refer to the HP, GP, UCP, and LCP because they all have heaters embedded into the plates for temperature control at high temperature. Normally, a metal of high thermal conductivity is suitable as a heater plate material because the surfaces of heater plates that face the sample specimen must have good temperature uniformity. A metering area is defined to quantify the amount of heat flow

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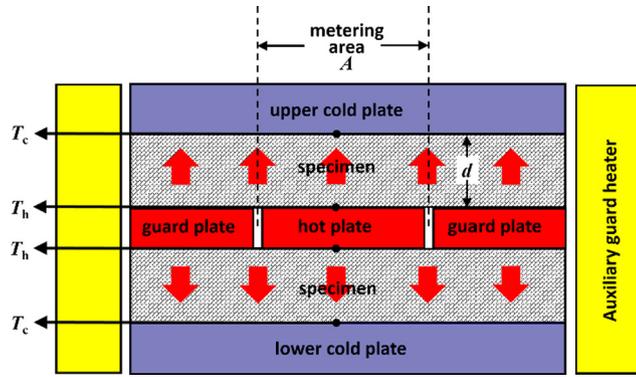


Fig. 1. General principle of the GHP apparatus with a double-sided configuration for measurement of the thermal conductivity of a sample specimen.

from the hot to the cold side of the specimen in a given area, and the boundary of the metering area is defined as the center of a low thermal conductivity gap (normally an air gap) between the HP and the GP. To ensure one-dimensional heat flow across the specimen, it is important that the temperature at the outer edge of the HP and the inner edge of the GP is controlled to be the same. Any deviation from it (called a ‘gap imbalance’) will cause lateral heat flow, and thus error in the measurement. If the width of the gap is too small, then even a small gap imbalance causes large lateral heat flow because of high thermal conductance across the gap. If the width is too large, then the one dimensionality of the heat flow is strongly distorted near the gap. Therefore, there is a proper gap width between the HP and the GP.

If the power dissipated from the hot side to the cold side across the specimen within the metering area  $A$  is measured as  $P$ , then the effective thermal conductivity  $\lambda$  of the sample specimen is

$$\lambda = \frac{P \cdot d}{2A \cdot \Delta T}, \quad (1)$$

where  $\Delta T = T_h - T_c$ , and  $d$  is the thickness of the specimen. There is a compromise in selecting a proper  $\Delta T$  in that a smaller  $\Delta T$  results in larger relative uncertainty in its measurement when assuming fixed accuracy in temperature measurements. On the other hand,  $\Delta T$  that is too large means that  $\lambda$  is not measured at exact temperature  $T_m$  if  $\lambda(T_m)$  is not linear for  $T_m$  in the temperature range between  $T_m - \Delta T/2$  and  $T_m + \Delta T/2$  because the specimen has a vertical temperature gradient from  $T_m - \Delta T/2$  to  $T_m + \Delta T/2$ .

The principles and robustness of the GHP method have been experimentally validated near ambient temperature over a long time period through comparisons of measurement results by GHP instruments of various constructions on the same specimen ([5] and references therein). A comparison of GHP measurements by the national measurement institutes of France and the USA for temperatures range from 7 to 47 °C showed agreement of within 1% [5]. Because the GHP method is robust and provides absolute measurement of thermal conductivity, it has also been used to certify standard reference materials (SRMs) for thermal conductivity measurements. The Institute for Reference Materials and Measurement provided the IRMM-440 reference which can be used as a thermal conductivity SRM in the range between –10 °C and 50 °C [6,7]. The thermal conductivity of this SRM is approximately  $0.030 \text{ W m}^{-1} \text{ K}^{-1}$  with an assigned relative uncertainty of 1% (with a 95% confidence interval). Meanwhile, the National Institute of Standards and Technology provided reference SRM 1450c that can be used between 7 °C and 67 °C with similar values of thermal conductivity and relative uncertainty [8]. Both SRMs can be used as reference materials for secondary thermal conductivity measurement methods such as the heat flow meter

or hot wire methods, or they can be used to verify newly built GHP apparatus.

The same principles behind GHPs near room temperature also applies to the measurement of thermal conductivity at high temperatures. However, developing a high temperature GHP apparatus is difficult because of several obstacles, including materials, heater life-time, electrical insulations, and design of the moving parts. Recently, there has been more research on the measurement of thermal insulation using the GHP method at high temperatures [9–12]. In a recent development, a metal alloy specimen with medium  $\lambda$  of  $\sim 10 \text{ W m}^{-1} \text{ K}^{-1}$  at temperatures up to 600 °C was measured with a high temperature GHP [9]. The researchers showed that measurements are more difficult for low thermal conductivity materials because the amount of intended one-dimensional heat flow is small, thus the effect of the unintended lateral heat flow becomes relatively larger. In another development, a high temperature GHP was constructed and results of thermal conductivity measurements up to  $T_m = 700 \text{ °C}$  were compared with results by various techniques including the GHP [10]. The estimated relative uncertainty at 700 °C was 9.4%, which is quite large. The new European Technical Specification CEN/TS 15548-1:2014 recognizes that it is unrealistic to expect a GHP measurement with an uncertainty of 2% for conductance measurements at temperatures above 100 °C [11]. The specification states that a realistic uncertainty is 5% up to 450 °C and 7% between 450 °C and 850 °C. Yet, in more recently developed GHP apparatus that operated up to 300 °C, the estimated uncertainty at 300 °C was 6.2% [12].

In the present work, we designed and constructed a GHP apparatus that can be used at high temperatures. The selection of materials, degree of thermal insulation to the ambient, power capacity of the heaters, and thermometers were designed so that the apparatus can be used at temperatures of up to 1000 °C. We describe details of the design parameters in the construction of the GHP apparatus, and preliminary test results of the apparatus with two specimens of thermal insulation materials are presented. There is no specified limit to the thermal conductivity of the specimen measured by this apparatus, but in the present work, the thermal conductivity of the specimens was between  $0.02 \text{ W m}^{-1} \text{ K}^{-1}$  and  $0.16 \text{ W m}^{-1} \text{ K}^{-1}$  at a temperature range of 100–800 °C.

## 2. Design and construction

Table 1 summarizes the design parameters of the high temperature GHP constructed in this work. In the following subsections, each parameter is described in detail.

### 2.1. Vacuum-tight chamber

We constructed the GHP apparatus in a cylindrical-shaped vacuum-tight chamber so that it was able to work in a controlled gas environment or under vacuum depending on the measurement requirements. A turbomolecular pump backed up by a mechanical pump whose capacity was 1000 L/min was installed to evacuate the chamber to high vacuum. However, during the preliminary measurements described in this work, only the mechanical pump was used by bypassing the turbomolecular pump. The measurements were performed in a nitrogen gas environment with pressure of approximately 100 kPa (i.e. one atmosphere). A digital pressure gauge was used to monitor the pressure inside the chamber. We started measuring at the lowest temperature (100 °C), then moved to higher temperatures, which means that the pressure increased inside the chamber as the temperature of the measurement was increased. Therefore, we relieved the pressure inside the chamber to near 100 kPa through a vent port before carrying out measurements at a given temperature.

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