Applied Thermal Engineering 62 (2014) 850–857

Contents lists available at SciVerse ScienceDirect

Applied Thermal Engineering

journal homepage: www.elsevier.com/locate/apthermeng

An optimal guarding scheme for thermal conductivity measurement using a guarded cut-bar technique, part 1 experimental study

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HIGHLIGHTS

- An optimal guarding scheme for the guarded cut-bar method was proposed.
- The influence of working condition on measurement accuracy was validated.
- In-situ particulate material effective thermal conductivity was measured.
- A general guideline for related methods requiring guarding was provided.

ARTICLE INFO

Article history:
Received 29 October 2012
Accepted 19 March 2013
Available online 6 April 2013

Keywords:
Thermal conductivity
Guarded cut-bar technique
Experimental measurement
Optimal guarding

ABSTRACT

In the guarded cut-bar technique, a guard surrounding the measured sample and reference (meter) bars is temperature controlled to carefully regulate heat losses from the sample and reference bars. Guarding is typically carried out by matching the temperature profiles between the guard and the test stack of sample and meter bars. Problems arise in matching the profiles, especially when the thermal conductivities of the meter bars and of the sample differ, as is usually the case. In a previous numerical study, the applied guarding condition (guard temperature profile) was found to be an important factor in measurement accuracy. Different from the linear-matched or isothermal schemes recommended in literature, the optimal guarding condition is dependent on the system geometry and thermal conductivity ratio of sample to meter bar. To validate the numerical results, an experimental study was performed to investigate the resulting error under different guarding conditions using stainless steel 304 as both the sample and meter bars. The optimal guarding condition was further verified on a certified reference material, pyroceram 9606, and 99.95% pure iron whose thermal conductivities are much smaller and much larger, respectively, than that of the stainless steel meter bars. Additionally, measurements are performed using three different inert gases to show the effect of the insulation effective thermal conductivity on measurement error, revealing low conductivity, argon gas, gives the lowest error sensitivity when deviating from the optimal condition. The result of this study provides a general guideline for the specific measurement method and for methods requiring optimal guarding or insulation.

1. Introduction

As a steady-state technique for thermal conductivity measurement, guarded cut-bar method [1–6] is ideal for measuring bar (long cylinder) shaped samples nondestructively. Studies of the cut-bar method are largely out-dated and absent from current literature as common thermal conductivity measurement techniques of study are frequently transient [7,8] or steady-state periodic [9,10] in nature using laser heating and/or detection. Such techniques have many advantages to classical steady-state techniques such as possibilities of being non-contact, fast, with very small spatial and temporal resolutions. Still, material characteristics (e.g. composites with rather large inclusions) or project-restricted requirements can require special geometries to be measured, restricting the selection of measurement techniques. The guarded cut-bar technique was selected as the measurement principle to design a system capable of measuring thermal conductivity of TRISO (tri-structural isotropic) fuel in its compact form (25 mm length × 12.3 mm diameter) to high temperatures (∼800 °C). Measurement is required to be nondestructive and the material

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http://dx.doi.org/10.1016/j.applthermaleng.2013.03.040

itself is a composite composed of millimeter-sized, multilayered particles embedded in a graphite matrix, thus requiring measurement to be made over the length of the compact. A detailed description of the measurement system design and associated determinate uncertainties may be found in Ref. [11].

The cut-bar technique consists of a test sample sandwiched between two reference comparators (meter bars) of known thermal conductivity comprising the sample column. The sample column is surrounded by insulation (vacuum in some cases [12]) which is encased in the guard. Temperature gradients can be controlled and measured in both the sample column and the guard. In this way, the heat flow through the measurement sample can be controlled to create known axial heat flow conditions for measurement of temperatures in the two reference comparators and the test sample. From the measured temperature gradients in the reference comparators, the heat flowing through the test sample can be calculated. With the heat flow and measured temperature gradient in the test sample, the thermal conductivity of the unknown material may be calculated.

The uncertainties and potential error sources associated with any measurement system and process should be analyzed extensively to improve accuracies and to optimize operation. In the evaluation of determinate uncertainty in the guarded cut-bar technique, the Taylor series method for propagation of uncertainty has been discussed by several references [11–13]. Investigating large discrepancies in the values of thermal conductivity of titanium carbide (TiC) reported by Vasilos et al. [14] and Taylor [15], Laubitz [16] evaluated systematic error caused by thermal conductivity mismatch between meter bars (standard) and specimen. Different from the typical, simplified “linear-matched guarding” in which the guard temperature matches that of the meter bars only at the top and bottom locations, Laubitz modeled a “continuous/overall matching condition” in which five independent guard heaters were employed to vary the temperature distribution. He concluded that if unknown specimen thermal conductivity varies greatly from that of the meter bars, very large systematic errors may result from this method.

In an analytical/numerical investigation of measurement error, Didion [17] based his results on two independent parameter types: conductivity and geometry factors. According to the evaluation of geometry effects (the test specimen geometry and the geometric relation between meter bars and specimen) and thermal conductivity effects (conductivity relations between meter bar, test specimen, and insulation material), he put forward a procedure for designing systems based on the cut-bar technique. Results of his analysis were adopted into the guidelines of an ASTM standard for this measurement method [18].

Using finite element analysis (FEA), a series of numerical simulations were recently carried out on error generation as a function of geometry, working conditions, as well as system component thermal conductivities [19]. The parametric studies on length ratio between specimen and sample column lengths, aspect ratio between specimen length and radius, insulation thermal conductivity effect, and the ratio of insulation layer thickness and specimen radius rendered conclusions similar to Didion’s analysis [17]. Analysis of thermal conductivity mismatch between meter bars and guard agreed with Laubitz’s conclusion [16]. The simulation of the mismatch ratio between specimen and meter bar radii coincided with the experimental observation by Babelot [20]. However, detailed studies on interfacial thermal resistance and guard/test-stack operating conditions (temperature gradients and average temperatures) have not been reported in literature.

Although the principles of measurement and heat transfer physics involved in the cut-bar technique are simple, careful implementation and understanding of the technique is required for good measurement results. Implementing a proper guarding scheme can potentially be challenging and complicated. The ASTM standard for the cut-bar technique recommends a linear matched or isothermal guarding profile, while also recommending careful system calibration when sample thermal conductivity varies much from that of the meter bars [18]. Therefore, the objective of this work consists of two parts: (i) the validation of the numerical simulation and (ii) the proposal of an “optimal guarding” scheme to simplify design and measurement of systems based on this technique. Using stainless steel 304 (SS304) as both the meter bars and the sample, the guard temperature gradient was adjusted to create the different working conditions discussed in Ref. [19]. Evaluation of the influence of other studied parameters including interfacial thermal contact resistance, average temperature difference, and nonlinear guard temperature gradient, was also conducted. The unequal average temperature levels of the guard and sample column are shown to have small effect on measurement results. Two samples whose thermal conductivities are much different from that of the meter bar were also measured. With the numerical simulation and experimental confirmation, the optimal guarding scheme is proposed. If the sample has a smaller thermal conductivity than the meter bar, the guard temperature gradient should be larger than that of linear match; whereas if the sample has a larger conductivity, the guard temperature gradient should fall between those of linear match and isothermal condition. Detailed guarding mechanism and means of determining and implementing the optimal guarding will be presented in part 2. The application of “optimum guarding” instead of “linear or overall matched guarding” is shown to primarily eliminate systematic errors associated with sample/meter bar thermal conductivity mismatch.

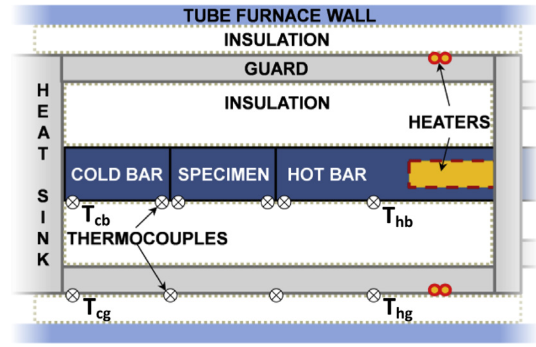

Fig. 1. Schematic illustration of the test section in a cut-bar measurement system.

2. Experimental description and numerical prediction

A detailed description of the measurement system and associated measurement uncertainties can be found in Ref. [11] and Fig. 1 presents a schematic of this technique. The thermal conductivity of the unknown specimen is determined by

$$q_{\mathrm{h,c}} = k_{\mathrm{m}}\left(\overline{T_{\mathrm{h,c}}}\right) \frac{\Delta T_{\mathrm{h,c}}}{\Delta z_{\mathrm{h,c}}} A_{\mathrm{m}} \tag{1}$$

$$k_{\mathrm{sc}} = \frac{q_{\mathrm{h}} + q_{\mathrm{c}}}{2A_{\mathrm{s}}} \frac{\Delta z_{\mathrm{s}}}{\Delta T_{\mathrm{s}}} \tag{2}$$

where q_h and q_c represent heat flow through hot and cold meter bars. ΔT_h [K], ΔT_c [K], ΔT_s [K], Δz_h [m], Δz_c [m] and Δz_s [m] are temperature differences and spacings for the meter bar and specimen shown in Fig. 1. k_m [W m^{-1} K^{-1}], A_m [m^2], k_{sc} [W m^{-1} K^{-1}] and A_s [m^2] are the thermal conductivities at mean temperature and