Title: Further Progress in the Development of a Slug Calorimeter for Evaluating the Apparent Thermal Conductivity of Fire Resistive Materials

Authors: Dale P. Bentz
Peter S. Gaal
Daniela S. Gaal

Published in: Thermal Conductivity 29/Thermal Expansion 17, Eds. J.R. Koenig and H. Ban, DesTech Publications, Inc., Lancaster, PA, 403-411, 2008.

ABSTRACT

A new method for evaluating the apparent thermal conductivity of fire resistive materials (FRMs) from room temperature to 750 °C using a "slug" calorimeter was presented in 2005. The continued development of this method is presented in this paper. A mini-furnace slug calorimeter experimental setup has been designed, constructed, and extensively employed to provide apparent thermal conductivities of a variety of FRMs. The development of an ASTM standard practice based on this measurement method is being pursued within the ASTM E37.05 Thermophysical Properties subcommittee. A preliminary evaluation of the single laboratory precision of the test practice has determined the precision to be ± 5 % below 500 °C and less than 10 % up to 750 °C. While the original version of the experimental setup employs twin specimens to produce an adiabatic boundary condition at the central plane of the steel slug, a single specimen version has recently been developed that relies on extensive insulation to produce an adiabatic boundary on the side of the steel slug not in contact with the test specimen. Computer modeling has been employed to demonstrate the validity of this approach. These efforts are all part of the ongoing "Performance Assessment and Optimization of Fire Resistive Materials" NIST/industry consortium that was initiated in March 2006.

INTRODUCTION AND BACKGROUND

Thermal conductivity of a fire resistive material (FRM) is a key thermophysical property in determining its ability to protect a (steel) substrate during a fire. A low thermal conductivity significantly slows the transmission of energy from the fire to the steel, prolonging the time before the mechanical properties of the steel will be significantly reduced, e.g., when its temperature exceeds about 500 °C (ASTM E 119, time-based performance rating to reach 538 °C). Because the temperatures

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Dale P. Bentz, Building and Fire Research Laboratory, National Institute of Standards and Technology, 100 Bureau Drive, Stop 8615, Gaithersburg, MD 20899-8615, U.S.A.
Peter S. Gaal, Anter Corporation, 1700 Universal Road, Pittsburgh, PA 15235-3998, U.S.A.
Daniela S. Gaal, Anter Laboratories, Inc., 1700 Universal Road, Pittsburgh, PA 15235-3998, U.S.A.

in a fire can exceed 1000 °C, the apparent thermal conductivity of the FRM must be determined at both room and elevated temperatures. FRMs present unique challenges to the measurement of thermal conductivity at elevated temperatures due to their inherent instability at high temperatures. These materials typically undergo phase changes, exothermic and endothermic reactions with significant changes in mass and dimensions, each of which can interfere with an accurate assessment of their thermal conductivity by steady state techniques.

With these limitations in mind, in 2004 a new technique was developed based on the utilization of a "slug" calorimeter to estimate the apparent thermal conductivity of an FRM during a dynamic multiple heating/cooling cycle experiment. The technique has been described in detail in a series of publications [1-3]. The underlying principles are similar to a transient test method originally described by Fitch [4] that is still utilized to estimate the thermal conductivity of leather [5]. As shown in Figure 1, a typical specimen design consists of a set of twin specimens of the FRM, each nominally 152 mm x 152 mm x 25 mm, placed in contact with the two surfaces of an AISI Type 304 stainless steel slug (152 mm x 152 mm x 12.7 mm). Twin specimens are employed to naturally produce an adiabatic boundary condition at the central plane of the slug. The steel slug contains three milled holes for the insertion of Type N thermocouples. The FRM/steel/FRM sandwich specimen is surrounded on four sides by a 25 mm thick microsilica high temperature guard insulation to produce a heat flow that is predominantly one-dimensional through the FRM specimens to the slug. A set of two Inconel retaining plates are used to maintain a slight compression on the specimens/guard insulation via a set of eight retaining bolts (two on each of the four edges of the plates). The experimental configuration is carefully placed in the center of a furnace, where it is subjected to a series of heating/cooling cycles. Both the slug and the outer FRM surface temperatures are monitored during these cycles to compute the apparent thermal conductivity of the FRM as a function of temperature as described below. By executing multiple heating/cooling cycles on the same specimens, the influences of endothermic (and sometimes exothermic) reactions and mass transfer of steam and other reaction gases on the apparent thermal conductivity can be determined [1-3].

The solution for determining the thermal conductivity from these temperature measurements has been derived in detail in reference [1]. When a steady heating or cooling rate, *F*, is applied, once transient effects subside, the apparent thermal conductivity at a given mean FRM specimen temperature can be determined by:

$$k = \frac{Fl\left(M_S c_p^S + M_{FRM} c_p^{FRM}\right)}{2A\Delta T} \qquad (1)$$

where k is the apparent thermal conductivity in units of [W/(m•K)]; F is the heating (or cooling rate) in units of K/s as measured for the steel slug, l is the specimen thickness in m; A is the cross-sectional area of the slug (or specimen, 0.152 m by 0.152 m = 0.0232 m^2 in our experimental setup); ΔT is the measured temperature difference across the FRM specimen; M_S and M_{FRM} are the masses of the steel slug and one of the (twin) FRM specimens in units of kg; and c_p^S and c_p^{FRM} are the

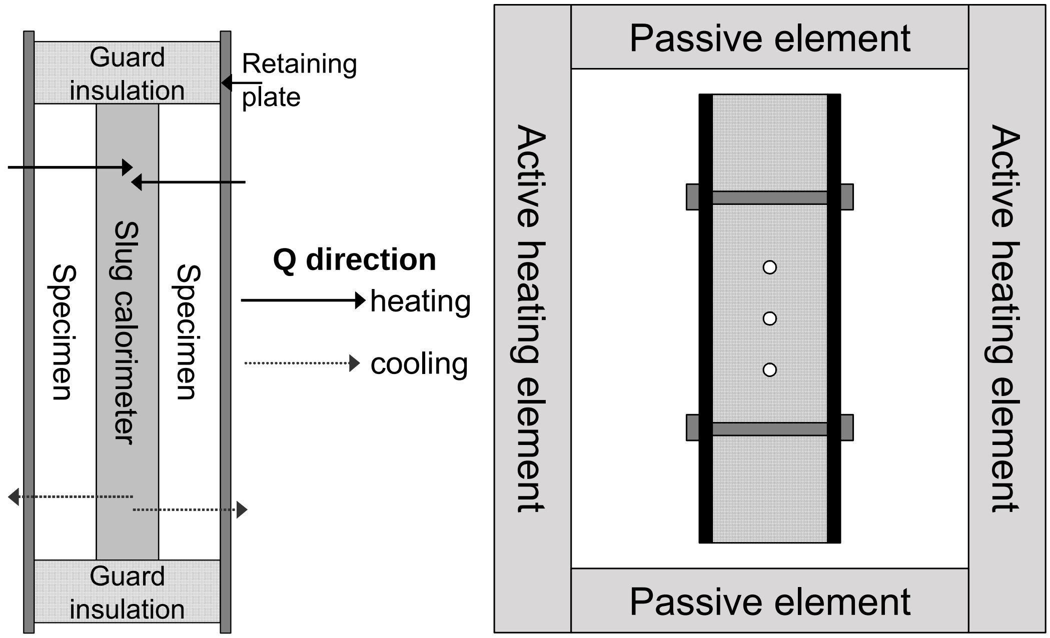

Figure 1. Schematics of the slug calorimeter test setup: left- schematic of a cross section through the middle of the basic slug calorimeter setup, and right- schematic of an overhead view of a completed sandwich specimen mounted and ready for testing in the mini-furnace.

temperature-dependent heat capacities of the steel plate and the FRM specimen in units of [J/(kg•K)]. The above solution has been derived assuming one-dimensional heat transfer and the maintenance of an adiabatic boundary condition at the central plane of the steel slug.

MINI-FURNACE SETUP

Originally, experiments were conducted in an electrically-heated box furnace with a working volume of 360 mm by 360 mm by 360 mm, a maximum operating temperature of 1773 K, and heating provided from exposed heating elements on all four sides of the interior. However, it soon became desirable to demonstrate that equivalent measurements could be achieved using a smaller furnace. The mini-furnace has a working volume of 250 mm by 250 mm by 300 mm and is constructed from a set of four ceramic fiber elements. As shown in Figure 1, only two of the elements contain active heating, while the other two function as passive insulators. The active heating elements measure 356 mm x 305 mm x 50 mm while the passive elements are 254 mm x 305 mm x 50 mm. The top and bottom of the furnace consist of 50 mm thick “plates” of the same high temperature insulation that is utilized as the guard insulation in the slug calorimeter. This mini-furnace has been used extensively over a period of more than nine months and the durability of both the ceramic fiber elements and the insulation boards has been good. The two active heating elements are connected to a control panel from which the temperature of the furnace can be programmed as a series of linear ramps, for example. Type N thermocouples used to monitor the temperatures of the outer FRM surface, the steel slug, and the furnace are connected to a simple USB-based data acquisition unit (with cold junction compensation) that can monitor up to eight channels simultaneously, and conveniently outputs the values into a spreadsheet.

Several comparisons were made between the original and mini furnaces to ensure compatible performance. Figure 2 shows the furnace temperatures achieved during a single heating/cooling cycle when both furnaces were programmed with the same set of piecewise linear temperature ramps. Specifically, the furnace setpoints were set to be 538 °C after 45 min, 704 °C after 70 min, 843 °C after 90 min, 927 °C after 105 min, and 1010 °C after 2 h. At the maximum points in the temperature/time curves shown in Figure 2, the furnaces were turned off and the temperatures continually monitored during natural cooling. For the minifurnace, results are shown for two different locations of the measurement thermocouple (37.5 mm and 87.5 mm from the heating element) while the control thermocouple was maintained at a distance of 37.5 mm in both cases, indicating the uniformity of the temperature distribution within the minifurnace. Due to its larger working volume, the original furnace exhibits a slight lag behind the temperature rise observed in the mini-furnace. Because the two furnaces are in different local environments, the mini-furnace being housed in a fume hood and the original furnace in a large open-bay laboratory, their cooling responses are also different. But, for all practical purposes, either furnace may be utilized to produce an acceptable heating/cooling curve for measuring apparent thermal conductivities of FRMs.

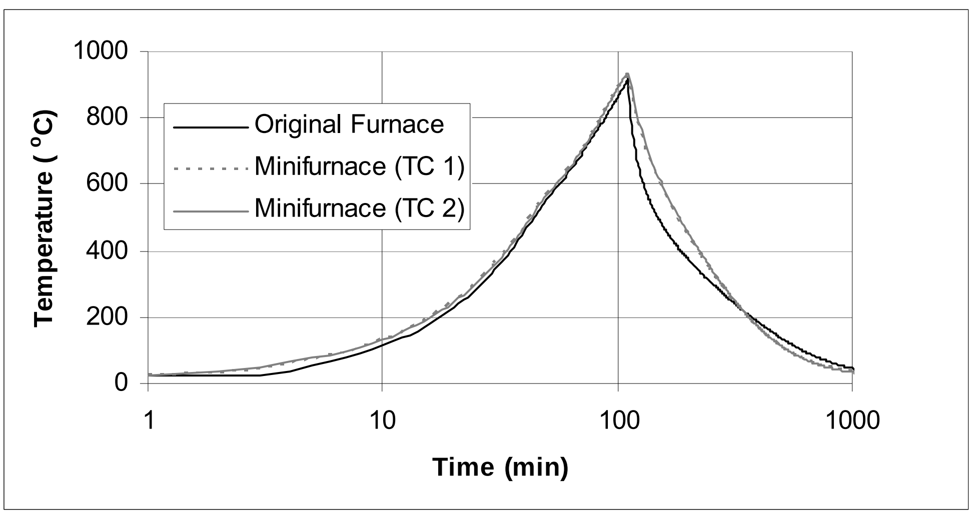

Figure 2. Comparison of measured furnace temperatures for the original and minifurnaces when programmed with the same set of linear temperature ramps.

One other difference between the two furnaces was noted during the course of these experiments. Because the heating elements in the original furnace are exposed, they will transfer considerable energy via radiation to the Inconel plates. Thus, at the later stages of the heating curve, as shown in Figure 3, the temperature of the outer surfaces of the FRM specimens actually may exceed that of the furnace

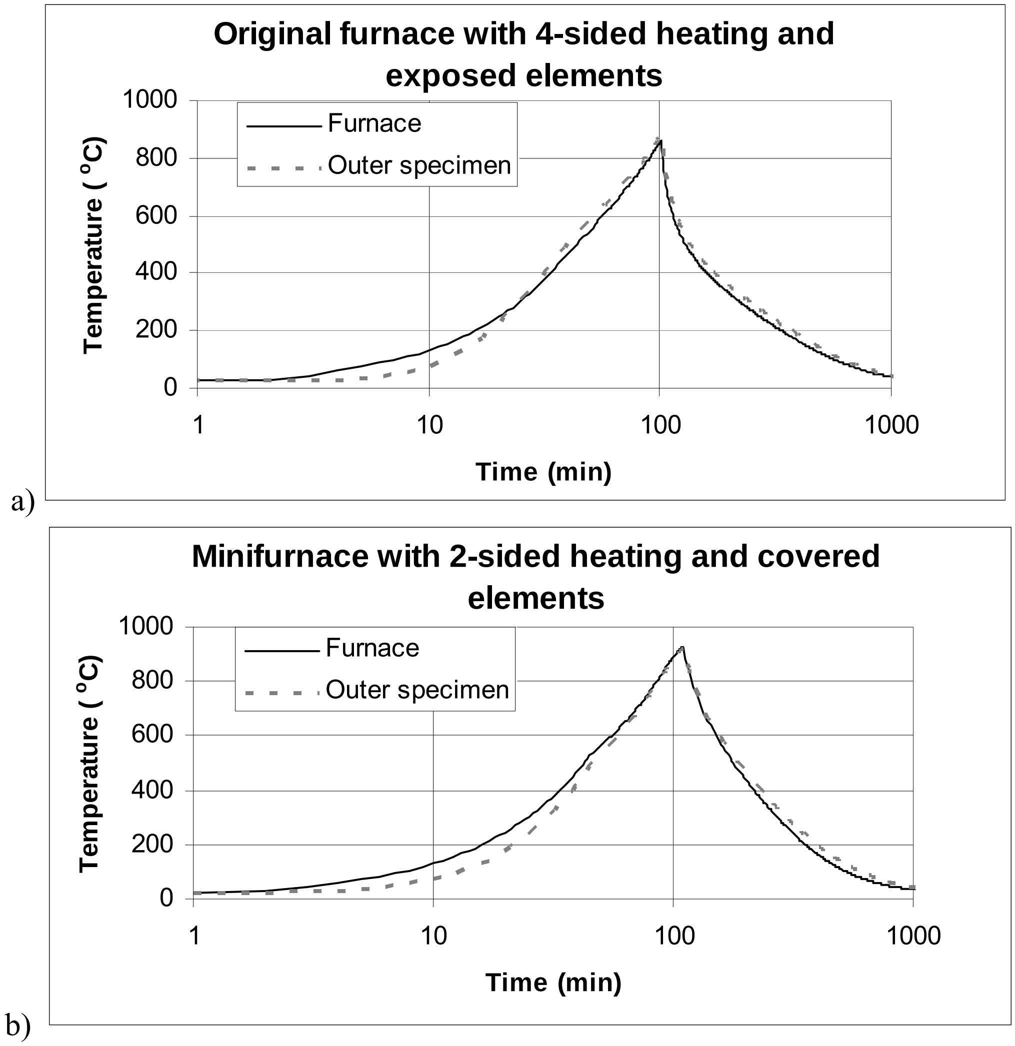

Figure 3. Comparison of measured furnace and outer FRM surface temperatures for the original and minifurnaces when programmed with the same set of linear temperature ramps.

environment. For the minifurnace, where the heating elements are embedded in the ceramic fiber boards, this effect is not observed, as the temperature of the outer FRM surface always remains below that of the furnace environment during heating (Figure 3). From a practical viewpoint, either of these cases is reasonable, as long as the temperatures of the steel slug and outer FRM surface are monitored throughout the heating/cooling cycles.

After it was verified that the two furnaces could produce similar heating/cooling curves, specimens of one commercial FRM were evaluated in both. As shown in Figure 4, the estimated apparent thermal conductivities for the two experiments are quite similar at temperatures below 400 °C, and are also in good agreement with a room temperature value provided by a transient plane source technique [3, 6] following the furnace exposure. At higher temperatures, the two specimens exhibit differences that are likely due to their differences in density. Although the same material composition was used in producing both sets of specimens, variability in the spraying process, curing, etc. resulted in their densities being different, as noted

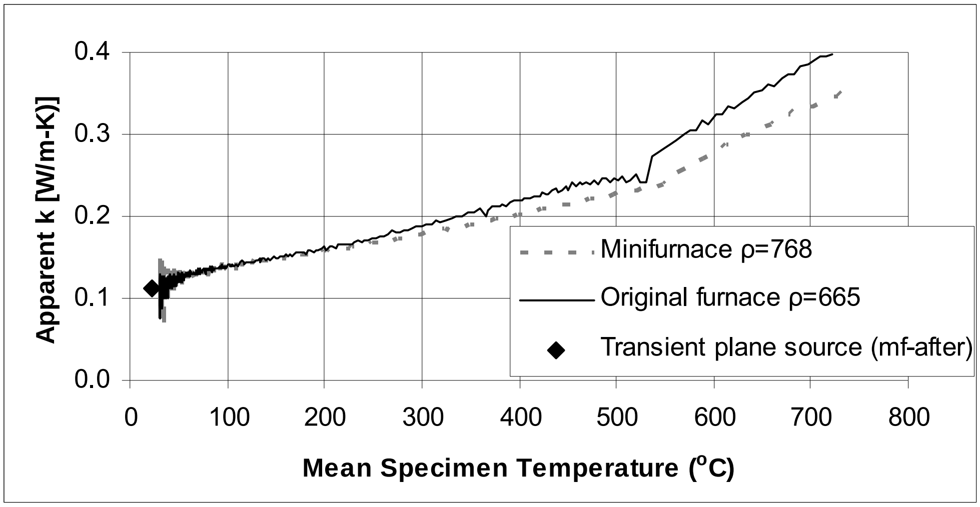

Figure 4. Comparison of apparent thermal conductivities determined during the 2nd heating/cooling cycle for separate specimens of a commercial fire resistive material in the original (large) and mini-furnace experimental setups. The original densities, ρ, of the two samples in kg/m^3 are indicated in the legend.

in Figure 4. These differences would especially influence the thermal conductivities at high temperatures due to enhanced radiative transfer in a lower density (more porous) material. This is indeed in agreement with the observed trends in Figure 4.

SINGLE SPECIMEN SYSTEM

From a practical standpoint, the construction of a sandwiched specimen assembly can be time consuming and can require a fair amount of skill. Since the ultimate purpose of the work is to produce a tool that is useful outside of the sheltered laboratory environment, some efforts were expended to simplify this task without impacting the overall usefulness of the method. A single sided configuration system was developed using one active specimen (unknown) and one dummy specimen on the other side of the slug, made from a very low thermal conductivity microsilica insulation material [7]. (This is a similar approach to what has been successfully used for single specimen guarded hot plate instruments.) The result is that the slug is permanently in place, encased in insulation, while heating is done only from one side.

In this case, equation (1) becomes:

$$k = \frac{Fl(M_S c_p^S + \frac{M_{FRM} c_p^{FRM}}{2})}{A \Delta T} \quad (2)$$

Using a heater identical to the ones in the mini-furnace, and after judicious scaling of the dummy specimen insulation with the aid of thermal modeling, the operating characteristics of the twin specimen system were duplicated, thus proving that either approach may be used for further work.

Figure 5 represents a computer model of the temperature distribution for a single specimen slug calorimeter system, where the back side of the steel slug is heavily insulated.

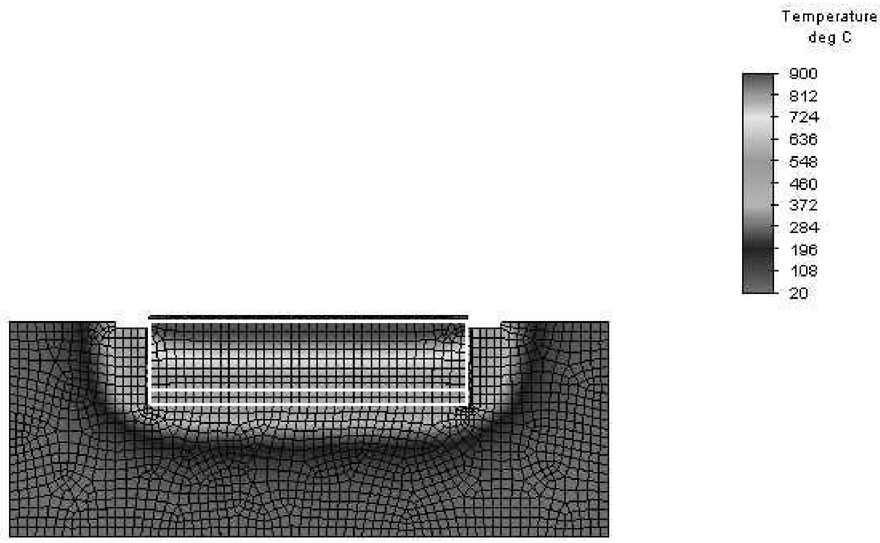

Figure 5. Computer model of the temperature distribution for a single specimen slug calorimeter.

The same types of materials were tested in the single specimen and in the twin specimen units, and the results obtained were within 5 % of one another [7].

ASTM STANDARDIZATION EFFORTS

The slug calorimeter test method is currently being balloted as a standard practice within the ASTM E37.05 Thermophysical Properties subcommittee [8]. The standard practice covers both the twin and single specimen configurations of the test method. To support development of the standard, replicate measurements were conducted on a series of five (twin) specimens of a single commercial spray-applied FRM with a nominal density of 450 kg/m^3. The results for the estimated apparent thermal conductivity as a function of mean specimen temperature are provided in Figure 6 for the values determined during the 2nd heating/cooling cycle of each individual experiment. Based on these results, a single laboratory precision statement was developed and incorporated into the draft standard. For the five specimens evaluated in the minifurnace at NIST, the determined coefficient of variation (the ratio of one standard deviation to the mean) was always less than 5 % for mean specimen temperatures of 500 °C or less, and less than 10 % for all temperatures up to 750 °C. Plans are currently being developed for an interlaboratory round robin to determine an appropriate interlaboratory precision statement to be incorporated into the standard practice.

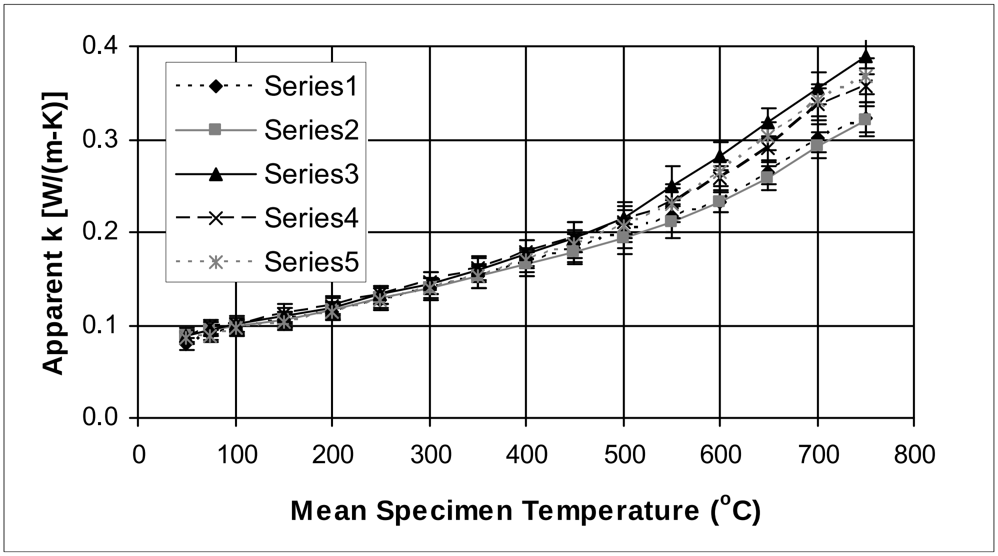

Figure 6. Comparison of apparent thermal conductivities determined during the 2nd heating/cooling cycle for five separate specimens of a commercial fire resistive material in the mini-furnace experimental setup. Error bars indicate estimated uncertainty in individual values as determined previously [1].

TRANSFER MATERIAL

Several users of the first generation slug calorimeters have expressed interest in having a transfer material that could be employed to verify the operational consistency of their equipment over time. A preliminary evaluation of the ability of a high strength, low density alumina refractory insulation [9] to serve this purpose has been performed. The insulation is rated for continuous use at temperatures up to 1800 °C. Testing in the slug calorimeter furnace has revealed basically no mass loss or dimensional changes during multiple heating/cooling cycles, as the material is pre-fired by its manufacturer. The material is available in 25.4 mm thickness sheets. Two 152.4 mm by 152.4 mm specimens were cut from such a sheet and their thermal conductivity estimated using the slug calorimeter technique. For this experiment, five separate runs of the slug calorimeter heating/cooling curves were conducted with the slug calorimeter experimental setup being totally disassembled and reassembled between the 2nd and 3rd runs. Experimental results are provided in Figure 7. The Figure 7 values for heating above 550 °C and cooling below 550 °C clearly define the slug calorimeter operation regions, i.e., after initial transients. The apparent thermal conductivities determined from the appropriate portions of both the heating ($T > 550$ °C) and cooling ($T < 550$ °C) curves are generally within 5 % of the mean values achieved during the five runs, indicating a potentially high stability for this proposed transfer material.

PROSPECTUS

Standardization of the slug calorimeter technique is proceeding. While much work remains to be done, the technique provides a novel method for evaluating the apparent thermal conductivity of fire resistive materials, and should be equally applicable to a wide variety of other (porous) solid materials with thermal conductivities in the range of 0.02 to 2.0 W/(m•K) [7].

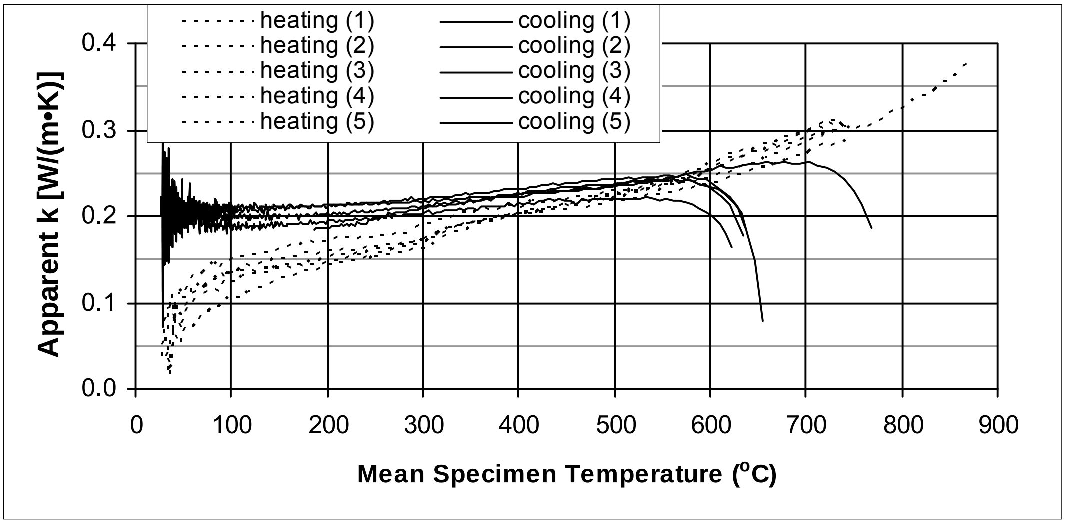

Figure 7. Comparison of apparent thermal conductivities determined during various heating/cooling cycle for specimens of a proposed reference material (alumina refractory) in the mini-furnace experimental setup.

ACKNOWLEDGEMENTS

The authors would like to thank Mr. Daniel Flynn of Metsys Corporation for his valuable contributions to the design and implementation of the slug calorimeter.

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