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Combination of Transient Plane Source and Slug Calorimeter Measurements to Estimate the Thermal Properties of Fire Resistive Materials*

ABSTRACT: The thermal properties of fire resistive materials (FRMs) as a function of temperature critically influence their ability to protect a (steel) structure during a fire exposure. Measurement of these properties is complicated by the microstructural heterogeneity of typical FRMs, the need to measure properties over a wide temperature range from room temperature to 1000°C and higher, and the reactions, phase changes, and volumetric changes that the materials may undergo during exposure to elevated temperatures. This paper presents an integrated approach for determining thermal properties via a combination of two experimental techniques, namely the transient plane source and the slug calorimeter methods. The former is utilized to provide an estimate of the volumetric heat capacity and a room temperature thermal conductivity value for the FRM under study, while the latter is employed to estimate the variation in effective thermal conductivity with temperature, including the influences of reactions and mass transport during multiple heating/cooling cycles. The combination of the two techniques is demonstrated for four different inorganic-based FRMs. Their extension to organic (intumescent) systems is also discussed.

KEYWORDS: building technology, fire resistive material, heat capacity, slug calorimeter, thermal conductivity, transient plane source technique

Introduction

Fire resistive materials (FRMs) have been utilized for many years [1] to protect steel (and other) substrates during a fire exposure, by limiting or reducing the temperature rise experienced by the steel. While it is obvious that the thermophysical properties, mainly thermal conductivity, heat capacity, density, and heats of reactions and phase changes of the FRMs will control their performance in this application [2], few standard test methods are readily available for actually measuring these properties over the wide temperature range of relevance during an actual fire exposure (from room temperature to greater than 1000°C, for instance). Most evaluations of these materials are currently performed on a pass/fail basis using the American Society for Testing and Materials (ASTM) Standard Test Methods for Fire Tests of Building Construction and Materials (E 119) [3], which provide a time-based performance rating (e.g., 1 h, 2 h, or 3 h, etc.). While these ratings are used daily by architects and designers for selecting passive fire protection strategies, they provide little direct information of value for engineers and scientists who are interested in simulating the fire/structural performance of buildings and other structures. While some 20 years ago, Wickstrom [4] first detailed how the thermal resistance of FRMs could be obtained from fire tests where furnace and steel temperatures were recorded, such computations are still rarely employed in the United States. The objective of this paper is to present a methodology for estimating the room temperature heat capacity and temperature variable thermal conductivity of FRMs by combining two existing experimental techniques, namely the transient plane source method [5,6] and a recently developed slug calorimeter technique [7]. In addition to providing estimates of these critical thermal properties, the presented protocol also provides supplemental information on the effects of chemical reactions, phase changes, and mass transport of reaction gases on the thermal performance of the FRMs being evaluated.

Experimental

Four inorganic FRMs were investigated, two each from two different manufacturers. Two (here denoted as A and B) are gypsum-based with different lightweight extenders (fillers) and two (denoted as C and D) are calcium silicate-based. The measured room temperature densities, heat capacities, and thermal conductivities of the four materials are provided in Table 1.

Specimens were nominally 152.4 by 152.4 by 25.4 mm, although the exact thickness of each specimen was assessed using a micrometre and averaging the measurements obtained from eight points (two coming in from each of the four edges). Following the physical characterization of the mass and dimensions of each specimen, thermal measurements were performed using either a Hot Disk Thermal Constants Analyzer (Uppsala, Sweden) for the transient plane source method or a recently constructed slug calorimeter [7]. For the transient plane source method, a nickel wire spiral probe with a radius of 14.67 mm was sandwiched between twin specimens of the FRM being evaluated, with a power input of 0.08 W for 320 s. The raw data were collected by the Hot Disk software analysis package and evaluated to provide estimates of the room temperature thermal conductivity and volumetric heat capacity of the FRM being evaluated. At least five separate room temperature measurements were made on each set of "twin" specimens and the average values are reported here. The average measured volumetric heat capacities were then converted to mass-based heat

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*Certain commercial products are identified in this paper to specify the materials used and procedures employed. In no case does such identification imply endorsement by the National Institute of Standards and Technology, nor does it indicate that the products are necessarily the best available for the purpose.

TABLE 1—*Thermophysical properties* (*with standard deviations*) *of FRMs.*

Specimen ID	Density, kg/m^3	Hot Disk Room Temperature Heat Capacity, $J/(kg \cdot K)$	Hot Disk Room Temperature Thermal Conductivity, $W/(m \cdot K)$	Mass Loss during Slug Calorimeter Testing, %
A	294±4.5	1220±10	0.120±0.001	21
B	367±1.5	1170±10	0.0983±0.0008	29
C	339±6.5	1100±10	0.0910±0.0003	15
D	506±1.2	1070±10	0.120±0.001	12

capacities [units of J/(kg·K)] using the measured room temperature average densities of the FRMs from Table 1. Hot Disk reports reproducibilities of ±2% for thermal conductivity and ±7% for heat capacity (specific heat per unit volume).

Separate (pairs of) specimens of each FRM were also evaluated in a high temperature electrical furnace using a simple slug calorimeter that has been described in detail previously [7]. Its underlying principles are similar to those in the apparatus originally described by Fitch [8] that is still utilized for estimating the thermal conductivity of leather in the ASTM Standard Test Method for Estimating the Thermal Conductivity of Leather with the Cenco-Fitch Apparatus (D 2214) [3]. The metal slug consists of an American Iron and Steel Institute (AISI) Type 304 stainless steel plate 152 by 152 by 12.7 mm containing three holes at the top for the insertion of high temperature Type N thermocouples [7]. The steel plate has a mass, M_S, of 2340 g, and the heat capacity as a function of temperature values for 304 stainless steel were taken from the literature [9]. With these thermophysical properties and the measured temperature of the steel slug as a function of time, the heat flow into the slug during heating (or from the slug during cooling) can be readily computed. Knowing the total heat flow (including the contribution to raising the temperature of the FRM specimens) and the temperature difference across the FRM, an effective thermal conductivity can be easily computed as outlined in [7] and using the following equation:

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

where k is the effective thermal conductivity [in W/(m·K)], F is the temperature increase (or decrease) rate (in K/s), l is the FRM specimen thickness (in m), M is mass (in kg), c_p is heat capacity [in J/(kg·K)], A is the FRM specimen area (152 by 152 mm), and ΔT is the temperature difference across the FRM specimen(s) (in K). In this paper, while the steel heat capacity as a function of temperature was included directly in the analysis, the FRM heat capacity needed for the slug calorimeter analysis in Eq. 1 will be supplied by the room temperature Hot Disk measurements and is assumed to be *constant* with temperature. While measurements conducted by a private laboratory [10] have indicated that the heat capacity of FRM A, for example, varies from 1046 $J/(kg \cdot K)$ to 1400 $J/(kg \cdot K)$ as temperature is increased from 50 to 600°C, using the single value of 1220 $J/(kg \cdot K)$ from Table 1 resulted in little visible difference in the calculated effective thermal conductivity curves, as the value of the $M_S c_p^S$ term in Eq. 1 is generally five to six times larger than that of the $M_{FRM} c_p^{FRM}$ term for the experimental setup employed in this study. Due to the heterogeneous microstructure of many FRMs it is often difficult to obtain a representative volume sample to determine their heat capacities using more conventional techniques such as the ASTM Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry (E 1269) [3]. Furthermore, for this study, the masses M_{FRM} of the FRM specimens were measured before and after the heating/cooling exposures in the furnace and the measured mass losses (values provided in Table 1) were distributed uniformly over the specimen temperature rise experienced during the first heating cycle.

A schematic and a photograph of the slug calorimeter experimental setup are provided in Fig. 1. While more information on the mathematical analysis and the expected uncertainties accompanying the slug calorimeter measurements can be found in Ref. [7], the most significant contributor to the expanded uncertainty is the uncertainty in the temperature measurement. For example, assuming an uncertainty of 1°C for the thermocouple readings and a 5 min sampling interval, the estimated uncertainty in the effective thermal conductivity would be about 5 % for values computed in the temperature range of 400 to 700°C during heating [7].

Results

The room temperature thermal conductivities and heat capacities measured by the transient plane source method for the four FRMs are provided in Table 1. For these four inorganic FRMs, there is only about 30 % variability among their room temperature thermal properties. As stated earlier, the room temperature heat capacity was utilized to determine an effective thermal conductivity as a function of temperature for the various slug calorimeter results. For example, slug calorimeter results for FRM A are presented in Fig. 2. In the two graphs in Fig. 2, results for three consecutive heating/cooling cycles are presented. For the first two heating curves, the furnace temperature (setpoint) was 538°C after 45 min, 704°C

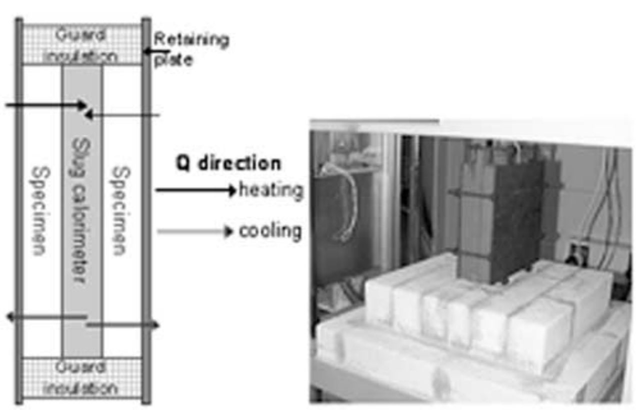

FIG. 1—*Schematic and photo of the slug calorimeter test setup:* (*left*) *schematic of a cross section through the middle of the basic slug calorimeter setup, and* (*right*) *photo of a completed sandwich specimen of a fumed-silica insulation board mounted and ready for testing in the box furnace.*

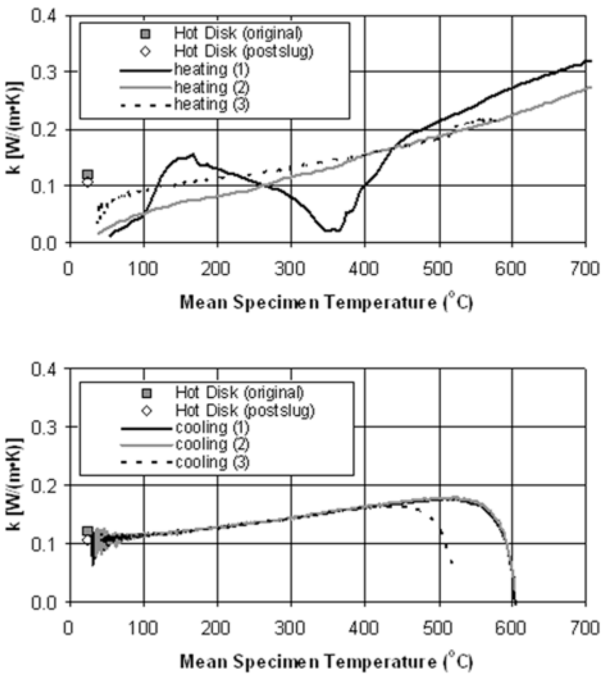

FIG. 2—*Effective thermal conductivity results for FRM A for multiple heating* (*top*) *and cooling* (*bottom*) *cycles in the slug calorimeter.*

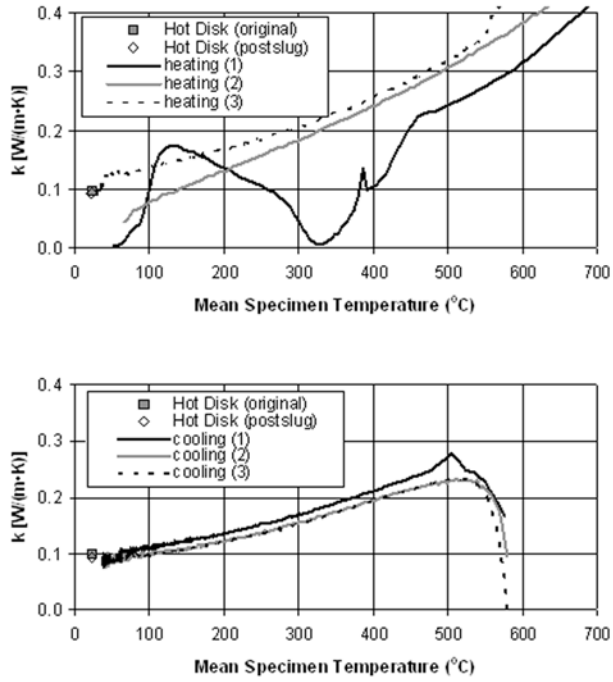

FIG. 3—*Effective thermal conductivity results for FRM B for multiple heating* (*top*) *and cooling* (*bottom*) *cycles in the slug calorimeter.*

after 70 min, 843°C after 90 min, 927°C after 105 min, and 1010°C after 2 h. For the third heating curve, the furnace temperature was raised linearly and much more gradually from room temperature to 600°C in 4 h, and then held constant for a period of time at 600°C, while the slug temperature gradually approached this value as well. All cooling curves were generated by simply turning off the furnace and monitoring the temperatures during its cooling back to room temperature.

Clearly, it takes a finite amount of time for the heat transfer to/from the slug to reach a pseudo-steady state during either initial heating or the transition from heating to cooling mode. Thus, the low temperature (<400°C) thermal conductivities for the heating curves and the higher temperature (>450°C) ones for the cooling curves should not be considered as providing useful effective thermal conductivity values, as the steady-state conditions assumed in the development of Eq 1 have not yet been achieved [7]. Still, the slug calorimeter may be utilized to provide estimates of the effective thermal conductivity from about 40 to 700°C by overlaying the heating ($\geqslant 400$°C) and cooling curves (<400°C) from the second heating/cooling cycle, for example [7]. Furthermore, by comparing the first and second heating curves, the influences of reactions and mass transport of reaction gases on the measured effective thermal conductivity can be examined. All four of the FRMs investigated in this study lose a finite mass, consisting mostly of water of hydration, during a high temperature exposure. While the initial decomposition of the hydrates and transformation from bound water to steam is endothermic (indicated by regions where the first heating cycle effective k falls below that of the second heating cycle), the subsequent (temperature and pressure driven) mass transport of the steam (and other hot reaction gases) towards the central steel slug appears as an "exothermic" component (indicated by the first heating cycle k exceeding the values determined from the second heating cycle). Generally, the "exothermic" behavior observed between 100 and 200°C would correspond to the mass transport of free water (steam), while that observed after about 400°C would correspond to water released from dehydration (of calcium sulfate dihydrate or hydrates of the calcium silicates). Separating the effects of these reactions from the "base" thermal conductivity of the material is necessary for detailed computational thermal performance modeling of these materials during an actual fire exposure [2], emphasizing the need to conduct multiple heating/cooling cycles during the slug calorimeter test.

Because these reactions generally go to completion during the first heating cycle, the effective k values for the three cooling curves basically overlap one another. As shown in Fig. 3, the one exception to this is found for FRM B where the first cooling curve differs significantly from the second and third ones. This FRM contained extruded polystyrene beads as its lightweight filler and when these beads decompose during the first heating cycle, they leave behind a series of relatively large pores that will influence both the thermal conductivity and dimensional stability of the FRM during subsequent heating and cooling cycles.

The effective thermal conductivity values determined at lower temperatures during the cooling cycles using the slug calorimeter can be compared to the values measured at room temperature, before and after the slug calorimeter test, using the transient plane source method. Results for the four different FRMs presented in Figs. 2–5 indicate a favorable comparison, particularly between the slug calorimeter cooling values and the transient plane source method values determined on the posttest FRM specimens. The transient plane source method values obtained on the original materials are always higher than those determined after testing in the slug calorimeter, likely due to the higher water contents in the original FRM specimens relative to those remaining in the specimens following multiple heating/cooling cycles in the slug calorimeter.

As indicated earlier in Table 1, the variability between room temperature thermal conductivities determined for the four FRMs examined in this study was only on the order of 30 %. However, as indicated in Figs. 2–5, at higher temperatures, this variability is

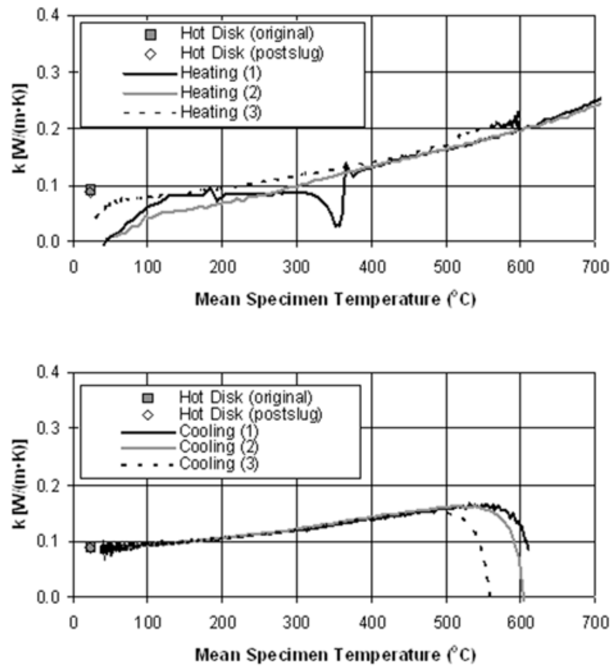

FIG. 4—*Effective thermal conductivity results for FRM C for multiple heating (top) and cooling (bottom) cycles in the slug calorimeter.*

much greater, approaching a value of 100 % when comparing the effective *k* values from the second heating curves at about 600 °C. Since it is these higher temperature effective thermal conductivities that will be of critical importance to the FRMs providing a thermal barrier and protecting the (steel) substrates during an actual fire, this study highlights the necessity of determining the thermal properties of FRMs over their expected in-use temperature range, and not just at room temperature.

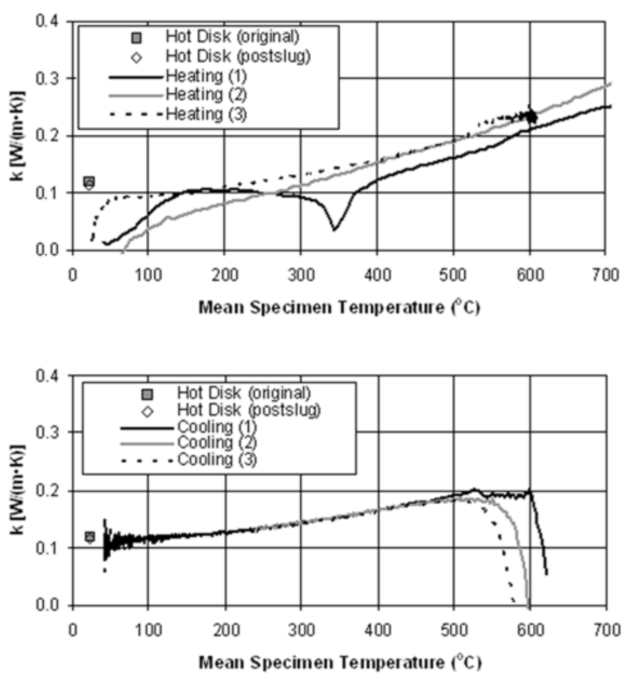

FIG. 5—*Effective thermal conductivity results for FRM D for multiple heating (top) and cooling (bottom) cycles in the slug calorimeter.*

In the present study, results for inorganic FRMs that undergo minimal dimensional changes during fire exposure have been presented. The extension to intumescent (organic or inorganic) materials that expand greatly (up to 40×) during a fire exposure will require modifications to the presented methodology. The transient plane source method can still be applied to determine the room temperature thermal conductivity and volumetric heat capacity, using the thin film module of the Hot Disk system, for example. For intumescent coatings, the slug calorimeter experimental setup has already been modified to contain retaining plates with a central square hole to allow for the (one-dimensional) expansion of the intumescent during the high temperature exposure [7]. However, to properly interpret the slug calorimeter results, detailed knowledge of these dimensional changes, as well as the energy transfer, during the test will be required. Options include using a (infrared) camera to monitor the expansion of the FRM in-situ or developing a (contact) sensor that can be placed in the experimental setup to provide continuous feedback on the "average thicknesses" of the twin FRM specimens.

Conclusions

An integrated approach to estimating the effective thermal conductivities of fire resistive materials has been presented. By combining room temperature transient plane source measurements with higher temperature slug calorimeter results, a more complete and coherent understanding of the thermal performance of these complex materials can be obtained. The influences of reactions and mass transport on the measured effective thermal conductivities and the need to make measurements at high temperatures have both been highlighted in this study.

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