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Determining thermal properties of gypsum board at elevated temperatures

Seul-Hyun Park[1], Samuel L. Manzello[1,*,†], Dale P. Bentz[2] and Tensei Mizukami[3]

[1]*Fire Research Division, Building and Fire Research Laboratory (BFRL), National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899 U.S.A.*
[2]*Materials and Construction Research Division, Building and Fire Research Laboratory (BFRL), National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899 U.S.A.*
[3]*Tsukuba Building Research and Testing Laboratory (TBTL), The Center for Better Living, Tsukuba, Ibaraki, Japan*

SUMMARY

The National Institute of Standards and Technology (NIST) and the Center for Better Living have formed a collaboration to assess the performance and failure mechanisms of gypsum wall assemblies under real fires/furnace conditions. These measurements are being used to compile an experimental database necessary to validate models that could be used to predict their performance and ultimate failure under various design fires. A critical component of the database is thermal property data of gypsum board. The present paper describes the results of an effort to quantify thermal properties of gypsum board. The thermal conductivity specific heat mass loss and linear contraction for gypsum board types widely used in the U.S.A. and Japan were measured both at room temperature and at elevated temperatures. The gypsum board types tested include Type X and Type C from the U.S.A. and Type R and Type F from Japan. Results indicate that the difference in thermal properties of all gypsum board samples tested in the present study is not significant particularly at elevated temperatures. A large difference in linear contraction among gypsum board samples was observed at elevated temperatures, implying a significant difference in mechanical behavior at fire temperatures. The experimental data set provides valuable information that can be used to model the behavior of gypsum board at elevated temperatures.

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KEY WORDS: gypsum board; thermal properties

INTRODUCTION

It is important to know when wall assemblies collapse and when their effectiveness as a smoke and flame barrier is compromised due to gypsum board shrinkage and cracking. Limited or no experimental data on the performance and failure mechanisms of gypsum board wall assemblies

*Correspondence to: Samuel L. Manzello, Fire Research Division, Gaithersburg, MD 20899 U.S.A.
†E-mail: samuel.manzello@nist.gov

under realistic fire loadings are available; this greatly hampers the application of performance-based design approaches [1–3]. Without physical knowledge of actual failure mechanisms, it is impossible to predict the time to failure of a gypsum board wall assembly under fire exposures using current methods. Such information is critical to determine safe egress times from buildings and provide guidance to firefighters entering a building.

To be able to model the behavior of gypsum board wall assemblies, thermal property data are needed as a function of temperature [1–4]. For gypsum board, critical data are either not available as a function of temperature or large uncertainties exist in regard to the data reported. Since a host of measurement methods have been used over the years to quantify gypsum board properties, it is very difficult to understand if differences in the reported measurements are due to materials properties or the varying methods employed. Properties of interest include specific heat and thermal conductivity as a function of temperature. In addition to these, the linear contraction and mass loss should be measured as a function of temperature for gypsum board. Furthermore, all of the aforementioned properties are needed for various gypsum board types.

The National Institute of Standards and Technology (NIST) and the Center for Better Living have formed a collaborative effort to assess the performance and failure mechanisms of gypsum wall assemblies under real fires/furnace conditions and to compile an experimental database necessary to validate models that could be used to predict their performance and ultimate failure under various design fires. Property determination is one important aspect of the data collection needed to be able to model the performance/failure of such assemblies; the results presented are part of a NIST effort to quantify gypsum board properties for various gypsum board types. The basic premise is to generate a database of these properties using a suite of in house metrology methods. This methodology will afford a uniform and consistent database for the needed properties necessary to model gypsum board assembly performance under a fire load.

To this end, these properties have been determined for common gypsum board types used in Japan and the U.S.A. These include Type X and Type C in the U.S.A.; Type R and Type F in Japan. The Hot Disk Thermal Constants Analyzer® (TPS 2500)[‡] was used to determine the room temperature thermal conductivity and specific heat of representative gypsum board samples, whereas the slug calorimeter and differential scanning calorimetry (DSC) were used to determine the thermal conductivity and specific heat as a function of temperature. For the mass loss and linear contraction measurements, a simultaneous measurement technique was developed with the aid of digital image processing software. Details of each measurement and the results are discussed and presented below. This paper greatly expands on a recently presented conference paper regarding gypsum board property determination [5].

EXPERIMENTAL DESCRIPTION THERMAL CONDUCTIVITY AND SPECIFIC HEAT MEASUREMENTS

The thermal properties of different types of gypsum board (Type X and Type C in the U.S.A.; Type R and Type F in Japan) were characterized both at room temperature and at elevated temperatures. All of the gypsum board samples were of the same nominal thickness; 15.9 mm. The Hot Disk

[‡]Certain commercial equipments are identified to accurately identify the methods used; this in no way implies endorsement from NIST.

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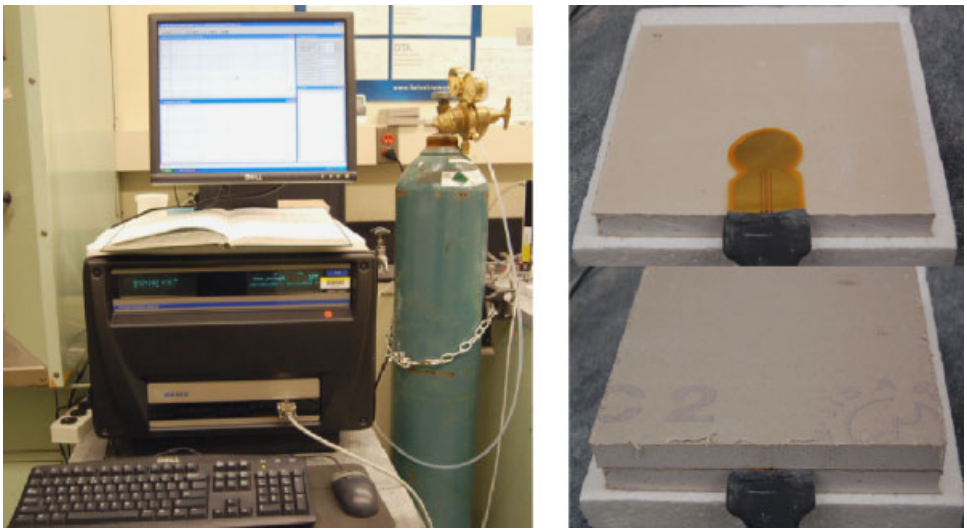

Figure 1. Configuration of a hot disk thermal constants analyzer; a spiral probe placed on the gypsum same is shown in the right.

Thermal Constants Analyzer® (TPS 2500) was used to determine the room temperature thermal conductivity and specific heat of representative samples of gypsum board. The Hot Disk determines thermal transport properties such as thermal conductivity and thermal diffusivity using the transient plane source technique (TPS). Briefly, a nickel wire spiral probe with a radius of about 15 mm was placed between two gypsum board samples, each with dimensions of 152 mm by 152 mm (see Figure 1). A constant current applied to the spiral probe creates resistance and thus increases the temperature of the spiral probe. The probe serves as the temperature sensor as well as the continuous plane heat source during the measurements. Since temperature changes in the probe are strongly dependent on sample composition, it is possible to evaluate the thermal transport properties of materials surrounding the probe. Based upon two calculated thermal transport properties, i.e. thermal conductivity and thermal diffusivity, heat capacity can be determined. As the Hot Disk measurement provides the volumetric heat capacity, the room temperature density was used to determine the specific heat on a mass basis.

To determine the specific heat as a function of temperature, DSC was used. DSC specific heat measurements were taken following the procedure outlined in ASTM E 1269-2001 [6]. The gypsum board samples used were 6–10 mg in initial mass. To accommodate the gas generation incurred from dehydration, the sample, reference and standard measurements utilized aluminum pans that were sealed except for a 50 μm pinhole in the lid. Measurements were performed with a heating rate of 20°C/min under a constant nitrogen gas flow. In addition, the specific heat of powdered sapphire Al_2O_3, as a correction material, was measured under the same operating condition used for the gypsum samples in order to obtain a correction factor. Details on this correction procedure are summarized in the ASTM E 1269-2001 [6].

Owing to the lack of an apparatus to measure continuous thermal conductivity versus temperature, previous determination of thermal conductivity of gypsum samples [4, 7] were performed at specified temperature intervals. Since such measurements require that the specimen is thermally

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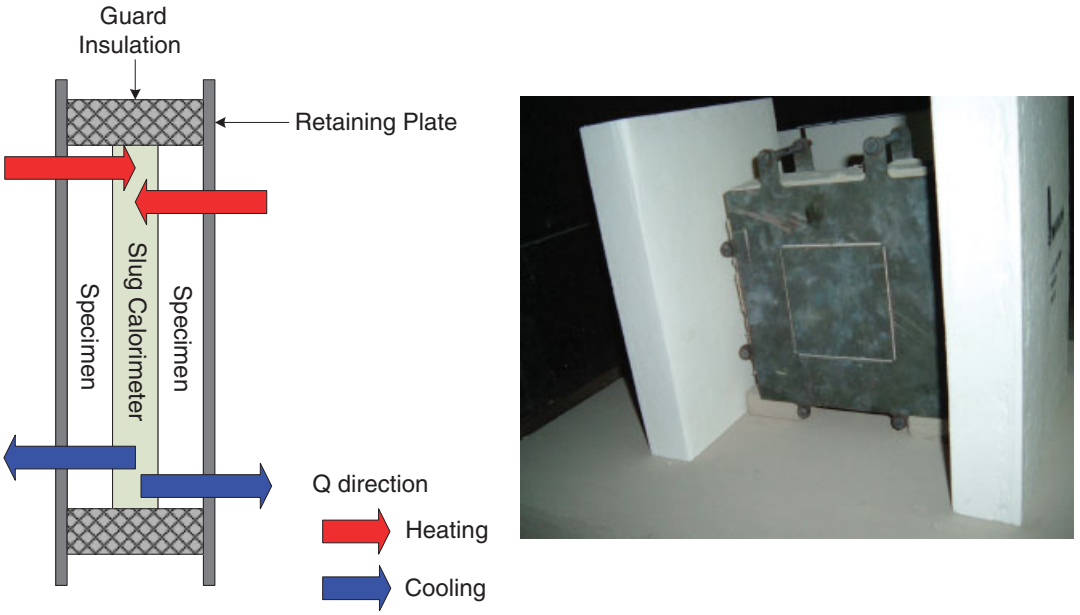

Figure 2. Schematic and picture of slug calorimeter.

equilibrated at test temperature prior to collecting the data, significant changes in the microstructure of the gypsum boards may occur, especially at high temperature, and result in potential errors in thermal conductivity determination. To minimize these practices, the thermal conductivity as a function of temperature was determined using the slug calorimeter [8] in the present study. This method utilizes continuous scanning to calculate the thermal conductivity. The slug calorimeter is composed of a square central stainless steel plate (152 mm by 152 mm by 12.7 mm). A set of 152 mm by 152 mm gypsum board samples (with their paper carefully removed) was installed in a 'sandwich' configuration (i.e. steel slug in the center); this provided an adiabatic boundary condition at the central axis of the slug plate. This entire configuration was then placed at the bottom of an electrically heated box furnace and the temperatures of the metal slug and exterior gypsum board surfaces were recorded during multiple heating and cooling cycles. Figure 2 displays a schematic of the slug calorimeter experimental setup. The steel plate has a mass of 2.3 kg and the heat capacity of stainless steel as a function of temperature was taken from the literature [9]. With knowledge of these properties and measured temperatures with time, an apparent thermal conductivity of the gypsum sample can be calculated using the following equation [8]:

$$k = \frac{Fl(M_{\mathrm{s}}C_{p.\mathrm{s}} + M_{\mathrm{g}}C_{p.\mathrm{g}})}{2A\Delta T} \tag{1}$$

where k is the apparent thermal conductivity, F is the temperature increase rate of the steel slug, l is the gypsum sample thickness, M_{s} and M_{g} are the masses of stainless steel and gypsum sample respectively, $C_{p.\mathrm{s}}$ and $C_{p.\mathrm{g}}$ are the heat capacity of stainless steel and gypsum sample, respectively, A is the gypsum sample area and ΔT is the temperature difference across the gypsum sample.

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In Equation (1), $C_{p,g}$ was assumed to be constant with temperature and determined using the Hot Disk measurement.

MASS LOSS AND LINEAR CONTRACTION MEASUREMENTS

To gain further insights into the physical behavior of gypsum board at elevated temperatures, the mass loss and linear contraction were measured as a function of temperature for the gypsum board samples. Triplicate samples of 15.9 mm thickness gypsum board were cut into rectangles of 152 mm by 50 mm from single sheets of each type and then placed into an oven. Fresh samples were heated up to 900°C for 3 h. Similar to prior work, it was observed that the additional mass loss beyond 3 h in the oven at a selected temperature was not significant [3]. This was verified by measuring the mass loss as a function of time (up to 24 h) at a given temperature. Consequently, after a 3 h heat-up at a selected temperature, samples were taken out to measure their mass loss and linear contraction. To aid in these measurements, a simultaneous measurement technique was developed. In this technique, the mass of each sample was simultaneously measured using a load cell with 0.01 g accuracy while a high resolution CCD camera imaged each sample placed on the scale. Figure 3(a) shows a schematic of the experimental setup for the simultaneous measurement. Each gypsum sample was recorded using a mini-DV recorder and the mass of each sample was saved using a user-developed lab view program before and after the heating procedure. This technique was different from prior work [3] and newly developed as part of this study.

The recorded gypsum sample images were digitized and analyzed using digital image processing software (Matrox, Inspector® 8.1). In addition, an analysis algorithm was developed and implemented to consistently interpret the digitized image of gypsum samples. In this algorithm, the background noise was first reduced using a 3×3 averaging filter and an edge-enhanced filter was applied to accentuate the edge features of each gypsum board sample. The image of the gypsum board samples was then extracted from the background by setting an appropriate threshold value. Eventually, the extracted images of each gypsum board sample were compared before and after the heating procedure to determine the linear contraction in the longitudinal direction (see Figure 3(b)).

GYPSUM BOARD PROPERTY CHARACTERIZATION

The thermal properties of four different gypsum boards types were characterized and compared (Type X and Type C in the U.S.A.; Type R and Type F in Japan). Table I displays the thermal properties of each gypsum sample obtained from the Hot Disk measurements at room temperature. These measurements were performed with the paper in place and with the paper removed from the gypsum board samples. Initially, for the Type X (U.S.A.) and Type C (U.S.A.) gypsum board samples, the paper was peeled off manually. As this technique was very laborious, an improved method was conducted for the Type F (Japan) and Type R (Japan) gypsum board samples. For these samples, the paper was removed by placing the gypsum board samples on a mill; this technique resulted in a reduction in thickness of 0.5 mm from each side of the gypsum board samples. The uncertainty in the measurement was found to be $\pm 10\%$. As summarized in Table I, the specific heat for all gypsum samples with the paper in place in the present study ranged from 891 J/(kg K) to 1017 J/(kg K); thermal conductivity varied from 0.254 W/(m K) to 0.314 W/(m K). The room temperature measurements were subsequently repeated with the paper removed. The removal of

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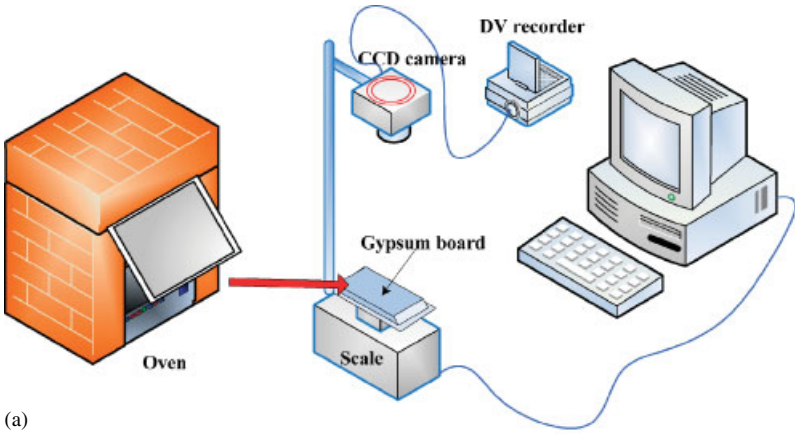
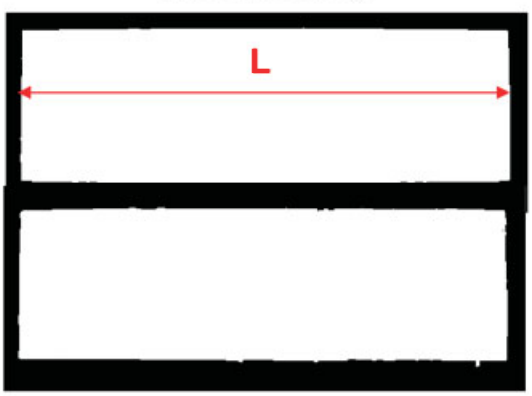


(a)

Images of Gypsum Samples Before and After Oven Drying



Thresholded Images of Gypsum Samples Before and After Oven Drying

(b)

Figure 3. (a) Drawing of experimental setup for mass loss and linear contraction measurements. (b) Images of gypsum board samples before and after oven drying; the threshold process for the images analysis technique is shown.

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Table I. Thermal properties of gypsum samples at room temperature (virgin material).

	With paper on		With paper off	
	C_p (J/kg K)	k (W/m K)	C_p (J/kg K)	k (W/m K)
Type C (U.S.A.)	1017	0.276	852	0.276
Type X (U.S.A.)	1089	0.258	947	0.252
Type F (Japan)	963	0.254	1034	0.238
Type R (Japan)	891	0.314	977	0.292

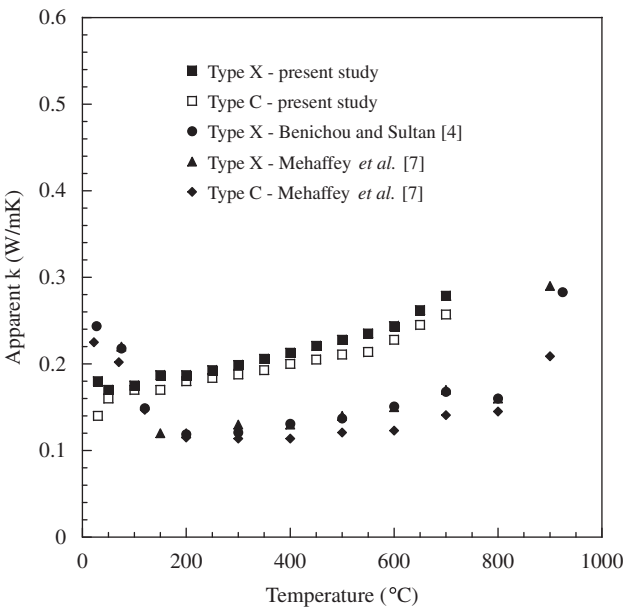

Figure 4. Thermal conductivity versus temperature (previously heated)—Type X (U.S.A.) and Type C (U.S.A.).

the paper influenced the C_p values. In addition, the room temperature density was determined for the gypsum board samples used. Including the paper, these values are: 711 kg/m^3 (Type X-U.S.A.); 752 kg/m^3 (Type C-U.S.A.); 743 kg/m^3 (Type F-Japan); 805 kg/m^3 (Type R-Japan). The uncertainty in density measurement was found to be $\pm 10\%$.

The thermal conductivity as a function of temperature was determined using the slug calorimeter [8] and the results are displayed in Figure 4. During the first heating cycle, the gypsum dehydrated, absorbed some of the energy, and delayed the temperature rise of the slug. To demonstrate these influences on the thermal conductivity measurement of gypsum samples, the thermal conductivities for the first and second heating cycles were calculated as a function of temperature and are displayed in Figure 5. The results clearly show that there are huge differences in the thermal conductivity between the first and second cycles for the temperature raging from 25 to 500°C. In the present study, the thermal conductivity was therefore determined based upon

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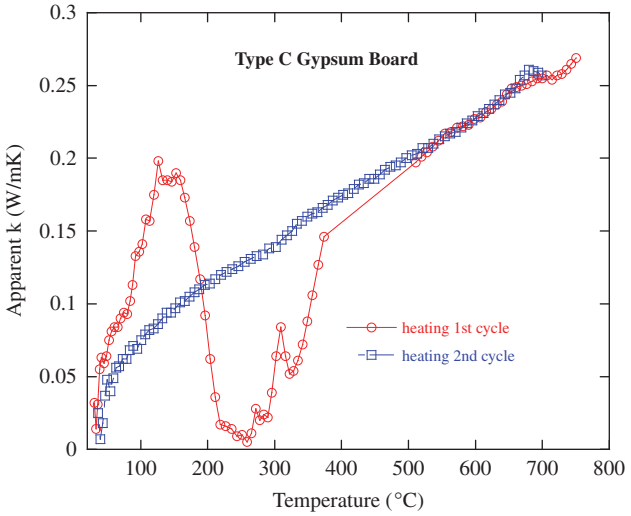

Figure 5. Thermal conductivity for Type C measured during the first and second heating cycles.

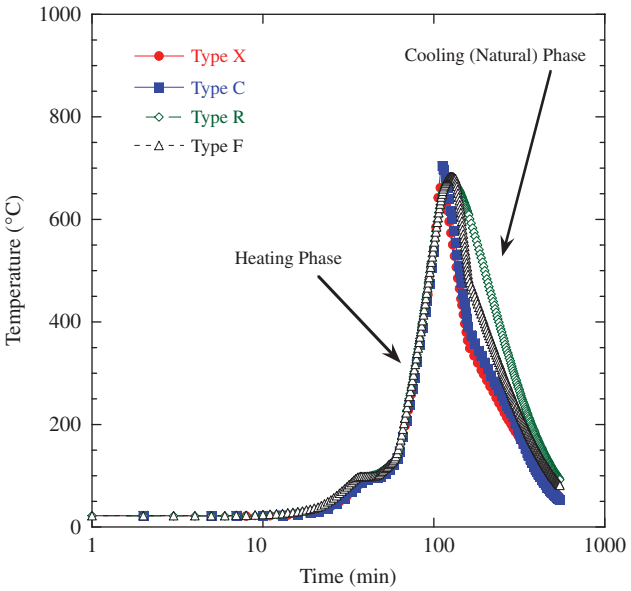

Figure 6. Average steel (slug) temperature as a function of time; three thermocouples were embedded inside the stainless steel slug.

the second heating/(natural) cooling cycle. For Type X (U.S.A.) and Type C (U.S.A.) gypsum board, the thermal conductivity steadily increased with temperature; similar behavior has been observed in thermal conductivity measurements for other gypsum board types [4]. There is a slight difference in thermal conductivity at low temperatures among the gypsum samples investigated. However, at elevated temperatures, the differences were minimal as shown in the figure.

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Bénichou and Sultan [4] measured thermal conductivity as a function of temperature for 15.9 mm Type X gypsum board; Type C board was not considered. In their work, a thermal conductivity meter was used. For comparison, at temperatures of 300 and 700°C, they reported values of 0.14 and 0.18 W/(m k) for Type X gypsum board, respectively. These values are slightly lower than the present measurements. Mehaffey *et al*. [7] also measured thermal conductivity for Type X and Type C gypsum board using a thermal conductivity meter and reported lower values of thermal conductivity at elevated temperatures as compared with the present measurements. For completeness, Figure 4 displays measurements conducted in the present study as compared with those of Bénichou and Sultan [4] and Mehaffey *et al*. [7]. Since the thermal conductivity meter employs a steady-state method to determine the thermal conductivity, the differences in the thermal conductivity between the present and previous measurements may be attributed to different measurement techniques used as well as the microstructure of the gypsum board which depends on composition, degree of crystallinity, average grain size, grain orientation, and porosity [7].

The thermal conductivity of the Type F (Japan) and Type R (Japan) gypsum board was not reported due to the large degree of cracking observed after the first heating and cooling cycle in the slug calorimeter for these materials. Figure 6 displays the steel slug temperatures measured during the first heating and cooling cycle. As can be seen, in contrast to the Type X (U.S.A.) and Type C (U.S.A.) gypsum board, during the first cooling cycle the temperature of the steel slug varied widely for the Japanese gypsum board measurements. The difference in temperature was caused by the severe cracking for Type R (Japan) and Type F (Japan) gypsum board (see Figure 7). Owing to more severe cracking behavior for Type R compared to Type F, the measured slug temperature for Type R shifted to higher regime as shown in the figure. This prevented an accurate measurement of thermal conductivity due to poor coverage of the slug by the gypsum board. Based on the steel slug temperatures measured during the first heating phase, it is apparent that the thermal conductivity is similar for all four gypsum board types. These results demonstrate that the use of the slug calorimeter for thermal conductivity measurements is limited for materials that crack severely during cooling.

Figure 8(a and b) displays the results of the DSC measurements for the Japanese and the U.S.A. gypsum board samples. The DSC traces demonstrate that two significant reactions are completed by the time that all samples reached 250°C. The core material of gypsum board is a porous solid composed primarily of calcium sulfate dihydrate ($CaSO_4 \cdot 2H_2O$), a naturally occurring mineral. The presence of the water molecules is a key feature in establishing the fire resistance properties of gypsum. When heated, crystalline gypsum dehydrates and water is liberated, typically in two separate, reversible chemical reactions [10]:

$$CaSO_4 \cdot 2H_2O + Q \leftrightarrow CaSO_4 \cdot (1/2)H_2O + (3/2)H_2O \quad (2)$$

$$CaSO_4 \cdot (1/2)H_2O + Q \leftrightarrow CaSO_4 + (1/2)H_2O \quad (3)$$

Both of these dehydration reactions are endothermic and generally occur at temperatures between 125 and 225°C.

In prior DSC measurements of gypsum board samples, Wakili and coworkers [11, 12] investigated the influence of the removal of the dehydration products on the specific heat using the different types of pans (which contain gypsum samples): (1) a pan without a lid, (2) a pan with a lid that had a 1 mm pin hole, and (3) a pan with a lid that had a 0.5 mm pin hole. In those measurements, a single broad peak was observed for experiments with the pan open (without the lid) while two peaks were observed for experiments with pans with the pinhole fitted to the lid.

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Figure 7. Cracking behavior of gypsum samples when heated at 700°C for Type R (Japan).

It was also found that the reduced pinhole size resulted in a slow removal of the dehydration product from the pan and led to a clear distinction between two dehydration reactions, similar to measurements reported in Figure 8(a) and (b).

In addition to two dehydration reactions, a third exothermic reaction occurs at a temperature of around 400°C (see Figure 8(a) and (b)), in which the molecular structure of the soluble crystal reorganizes itself into a lower insoluble energy state (hexagonal to orthorhombic):

$$\mathrm{CaSO_4(sol)} \rightarrow \mathrm{CaSO_4(insol)} + Q \tag{4}$$

As displayed in the figures, Type X gypsum board (U.S.A.) and Type R gypsum board (Japan) have C_p peaks of similar magnitude, which indicates the energy needed for dehydration (heat of reaction) was quite similar. In addition, the magnitudes of C_p peaks for Type C gypsum board (U.S.A.) were comparable to those of the C_p peaks for Type F gypsum board (Japan). The authors are not aware of specific heat data as a function of temperature for Type F (Japan) and Type R (Japan) gypsum board. For Type X 15.9 mm gypsum board, Bénichou and Sultan [4] reported specific heat measurements as a function of temperature using DSC methods. In those measurements, only the first dehydration reaction was observed; namely the reaction described in Equation (2) was not observed. With regard to the magnitude of the C_p peak, Bénichou and Sultan [4] reported a C_p value of 28 000 J/(kg K) at a temperature of 125°C. This is slightly higher than the reported values in the present study (see Figure 8(a)).

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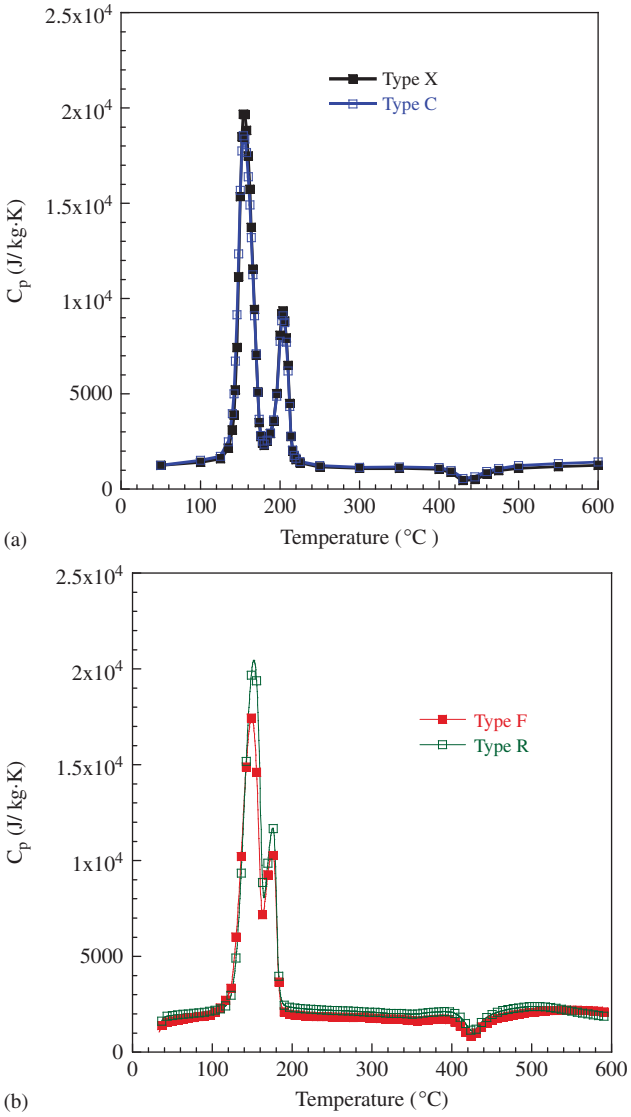

Figure 8. (a) Specific heat versus temperature—Type X (U.S.A.) and Type C (U.S.A.). (b) Specific heat versus temperature—Type F (Japan) and Type R (Japan).

The mass loss was measured for all gypsum board samples and is plotted as a function of temperature in Figures 9(a) and (b), respectively. At each temperature, each data point represents the average of three replicate measurements. As can be seen in Figure 9(a), a significant amount of mass loss was observed for all gypsum board samples for temperatures up to 400°C. This result is expected since the dehydration reactions are completed at temperatures above 250°C. The Type C gypsum board (U.S.A.) and Type F gypsum board (Japan) were similar in terms of the temporal variation in mass loss. The temporal variation in mass loss behavior was also similar for Type X

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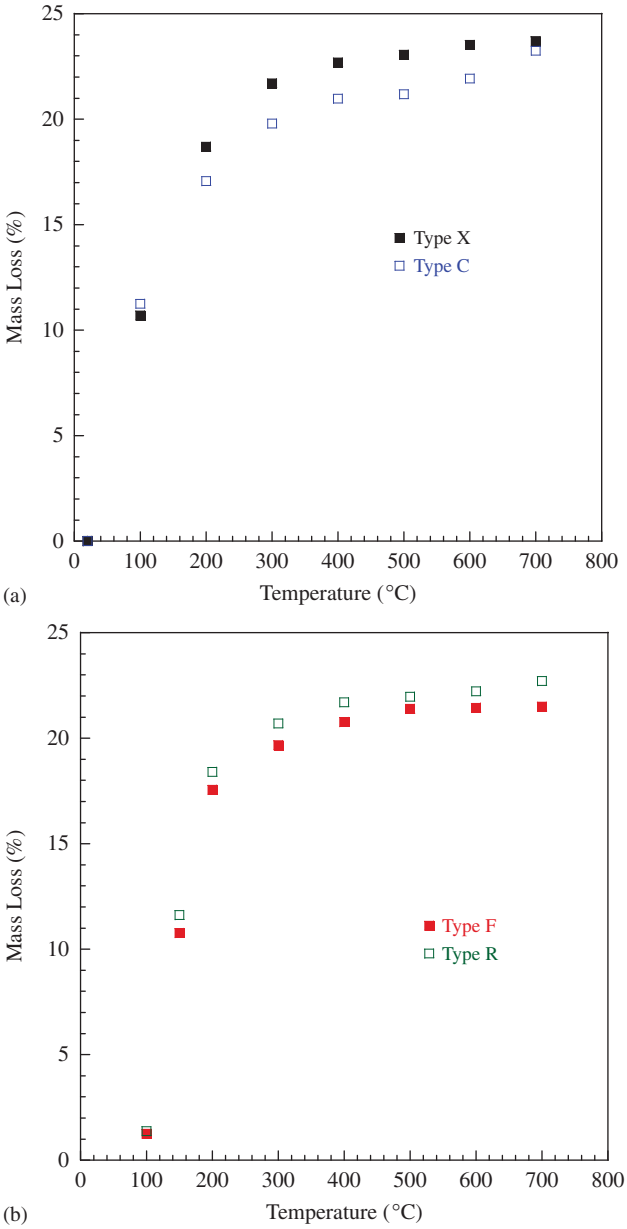

Figure 9. (a) Mass loss versus temperature—Type X (U.S.A.) and Type C (U.S.A.). (b) Mass loss versus temperature—Type F (Japan) and Type R (Japan).

gypsum board (U.S.A.) and Type R gypsum board (Japan). Differences in the mass loss observed between two groups may be due to the composition of the materials of each gypsum type which are added for fire resistance characteristics.

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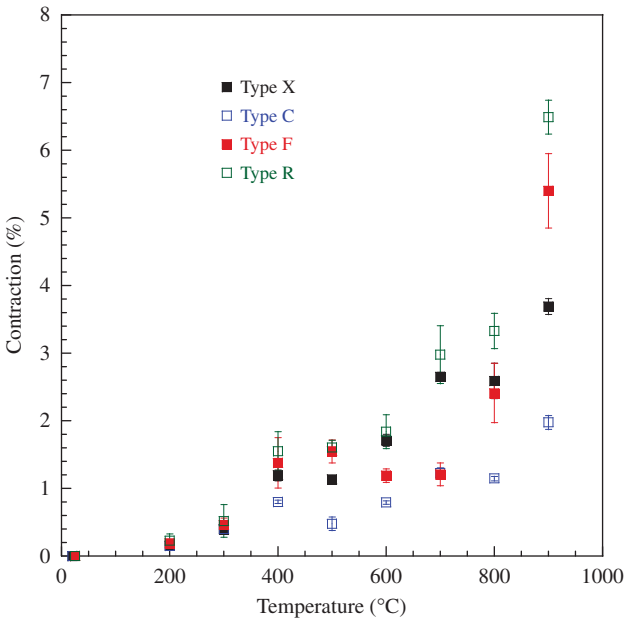

Figure 10. Linear contraction versus temperature-Type X (U.S.A.), Type C (U.S.A.), Type F (Japan), and Type R (Japan).

The linear contraction of all gypsum board samples is displayed in Figure 10. At each temperature, each data point represents the average of three replicate measurements. The contraction of each gypsum board sample was negligible at temperatures up to 300°C. On the other hand, differences in the contraction of each gypsum type were found to be considerably significant at higher temperatures. These results suggest that the mass loss due to the dehydration reactions has little effect on contraction of each gypsum sample. Data are available in the open literature for the linear contraction of 12.7 mm Type X gypsum board. Takeda [13] measured the linear contraction of 50 mm by 200 mm by 12.7 mm thick Type X gypsum board and reported that the contraction increased as a function of temperature; 1.7% at a temperature of 700°C. While the contraction measured by Takeda is lower than the reported values for Type X board here, the thickness of the board is different which should influence the results.

Clearly, the linear contraction of the gypsum board is strongly dependent on the composition of the additives. Common additives used to mitigate contraction of the boards include vermiculite. The present results suggest that Type C (U.S.A. board) contains the highest degree of additives as compared with the other board types tested. In addition to this, NIST is currently determining mechanical properties of the various gypsum board types as a function of temperature; this work will be the subject of future publications.

CONCLUSIONS

To be able to model the behavior of gypsum board wall assemblies, thermal property data are needed as a function of temperature. For gypsum board, critical data are either not available as a

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function of temperature or large differences exist in the data reported. Properties of interest include specific heat, density, and thermal conductivity as a function of temperature. The results presented are part of a NIST effort to quantify gypsum board properties for various gypsum board types.

The thermal conductivity specific heat mass loss and linear contraction for gypsum board types widely used in the U.S.A. and Japan were measured both at room temperature and elevated temperatures. Results indicate that the difference in specific heat of all gypsum board samples tested in the present study is not significant particularly at elevated temperatures. A large difference in linear contraction among gypsum samples was observed at elevated temperatures. The experimental data set provides valuable information that can be used to model the behavior of gypsum board at elevated temperatures. As part of the database for gypsum board, NIST is currently determining mechanical properties of various gypsum board types; such work will be the subject of future publications. Finally, it is desired to characterize other gypsum board types in addition to those used in Japan and the U.S.A.

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