

MEASUREMENT OF THERMAL PROPERTIES OF GYPSUM BOARD AT ELEVATED TEMPERATURES

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ABSTRACT

The thermal conductivity, specific heat, mass loss, and linear contraction for gypsum board types widely used in the USA and Japan were measured both at room temperature and elevated temperatures. The gypsum board types tested include Type X and Type C from the USA 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.

1. INTRODUCTION

For a performance-based design approach, 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 under realistic fire loadings are available; this greatly hampers the application of performance-based design approaches¹⁻³. Furthermore, to be able to model the behavior of gypsum board wall assemblies, thermal property data are needed as a function of temperature¹⁻⁴. For gypsum board, critical data is either not available as a function of temperature or large uncertainties

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exist in regard to the quality of the data reported. 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.

These properties have been determined for common gypsum board types used in Japan and the USA. These include Type X and Type C in the USA; Type R and Type F in Japan. The collaboration between the National Institute of Standards and Technology (NIST) and the Center for Better Living is part of an international 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, 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 aid of digital image processing software. Details of each measurement and the results are discussed and presented below.

2. EXPERIMENTAL DESCRIPTION

2.1 Thermal Conductivity and Specific Heat Measurements

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

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

To determine the specific heat as a function of temperature, differential scanning calorimetry (DSC) was used. DSC specific heat measurements were taken following the procedure outlined in ASTM E 1269-2001⁵. 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 $^{\circ}\text{C}/\text{min}$ 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 1269-2001⁵.

The thermal conductivity as a function of temperature was determined using the slug calorimeter⁶. The slug calorimeter is comprised 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 1 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 literature⁷. With knowledge of these properties and measured temperatures with time, an apparent thermal conductivity of the gypsum sample can be calculated using the following equation⁶:

$$k = \frac{Fl(M_s C_{p,s} + M_g C_{p,g})}{2A\Delta T} \quad (1)$$

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

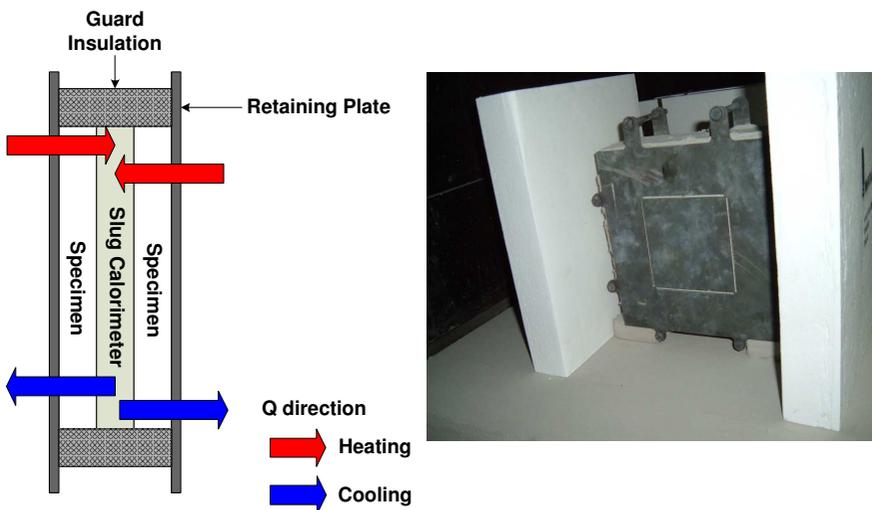


Fig. 1 Schematic and picture of slug calorimeter

2.2 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 three hours in the oven at a selected temperature was not significant³. This was verified by measuring the mass loss as a function of time (up to 24 hours) at a given temperature. Consequently, after a three hour 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 2 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 work³ and newly developed as part of this study.

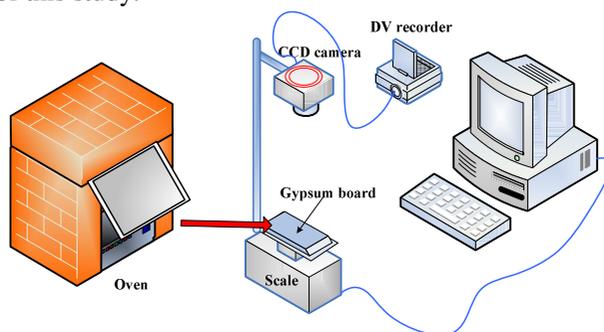


Fig. 2 Schematic of experimental setup for mass loss and linear contraction measurements

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 by 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 were 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.

3. GYPSUM BOARD PROPERTY CHARACTERIZATION

The thermal properties of four different gypsum boards types were characterized and compared (Type X and Type C in the USA; Type R and Type F in Japan). Table 1 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 (USA) and Type C (USA) 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 1, 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 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/m³ (Type X-USA); 752 kg/m³ (Type C-USA); 743 kg/m³ (Type F-Japan); 805 kg/m³ (Type R-Japan). The uncertainty in density measurement was found to be $\pm 10\%$.

Table 1 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 (USA)	1017	0.276	852	0.276
Type X (USA)	1089	0.258	947	0.252
Type F (Japan)	963	0.254	1,034	0.238
Type R (Japan)	891	0.314	977	0.292

The thermal conductivity as a function of temperature was determined using the slug calorimeter⁶ and the results are displayed in Figure 3. During the first heating cycle, the gypsum dehydrated, absorbed some of the energy, and delayed the temperature rise of the slug. As a result, the thermal conductivity was determined based upon the second heating/(natural) cooling cycle. For Type X (USA) and Type C (USA) gypsum board, the thermal conductivity steadily increased with temperature; similar behavior has been observed in thermal conductivity measurements for other gypsum board types⁴. 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. Bénichou and Sultan⁴ 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 °C and 700 °C, they reported values of 0.14 W/(m k) and 0.18 W/(m k) for Type X gypsum board, respectively. These values are slightly lower than the present measurements.

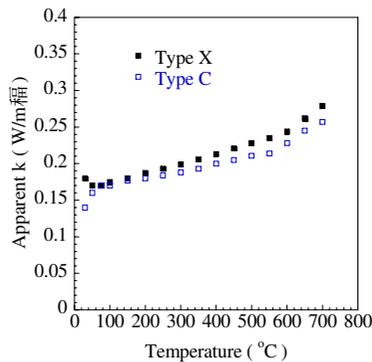


Fig. 3 Thermal conductivity vs. temperature (previously heated) -Type X (USA) and Type C (USA)

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 4 displays the steel slug temperatures measured during the first heating and cooling cycle. As can be seen, in contrast to the Type X (USA) and Type C (USA) 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. 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.

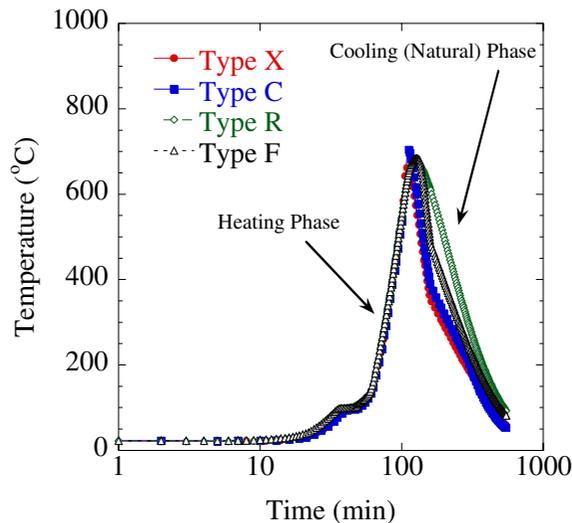
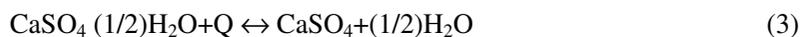


Fig. 4 Average steel (slug) temperature as a function of time; three thermocouples were embedded inside the stainless steel slug

Figure 5a-b displays the results of the DSC measurements for the Japanese and USA gypsum board samples. The DSC traces demonstrate 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 ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), 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 reactions⁸:



Both of these dehydration reactions are endothermic and generally occur at temperatures between 125 °C and 225 °C. At a temperature of around 400 °C, a third, exothermic reaction occurs, in which the molecular structure of the soluble crystal reorganizes itself into a lower insoluble energy state (hexagonal to orthorhombic):



As displayed in the figures, Type X gypsum board (USA) 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 (USA) 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 Sultan⁴ 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 Sultan⁴ 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 Fig. 5a).

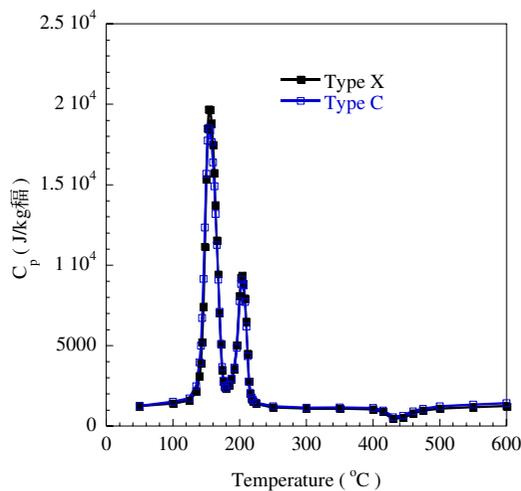


Fig. 5a Specific heat vs. temperature-Type X (USA) and Type C (USA)

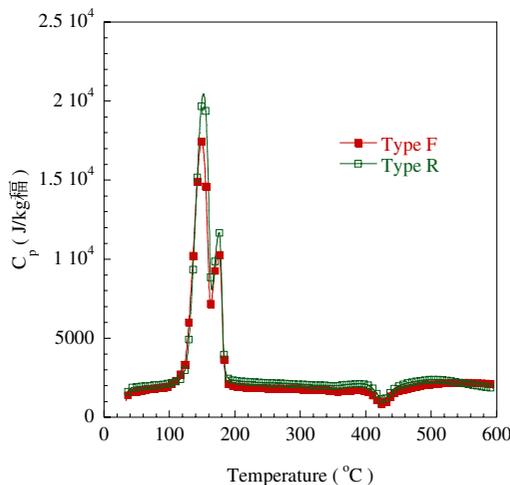


Fig. 5b Specific heat vs. temperature-Type F (Japan) and Type R (Japan)

The mass loss was measured for all gypsum board samples and is plotted as a function of temperature in Figures 6a and 6b, respectively. At each temperature, each data point represents the average of three replicate measurements. As can be seen in Figure 6a, 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 (USA) 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 gypsum board (USA) 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.

The linear contraction of all gypsum board samples is displayed in Figure 7. 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 is available in the open literature for the linear contraction of 12.7 mm Type X gypsum board. Takada⁹ 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 Takada 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 (USA board) contains the highest degree of additives as compared to 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.

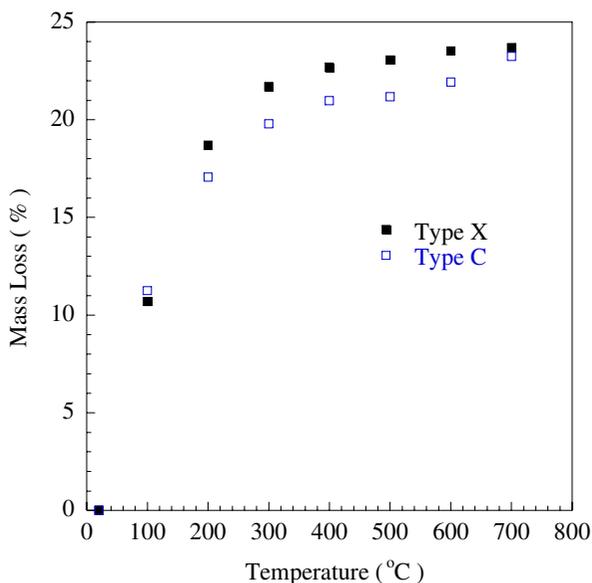


Fig. 6a Mass loss vs. temperature-Type X (USA) and Type C (USA)

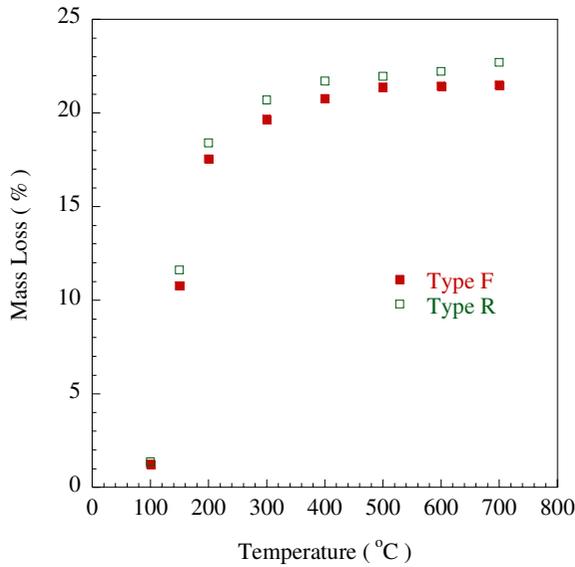


Fig. 6b Mass loss vs. temperature-Type F (Japan) and Type R (Japan)

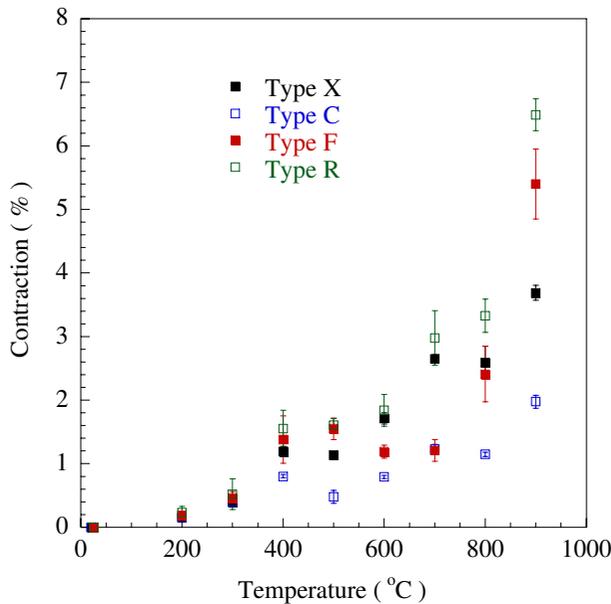


Fig. 7 Linear contraction vs. temperature-Type X (USA), Type C (USA), Type F (Japan) and Type R (Japan)

4. 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 is either not available as a function of temperature or large uncertainties exist in regard to the quality of

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 USA 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 USA.

5. ACKNOWLEDGMENTS

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