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An Initial Investigation of the Properties and Performance of Magnesium Oxychloride-Based Foam Thermal Insulation

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ABSTRACT

This study is an initial investigation of the properties and performance of magnesium oxychloride-based foam thermal insulation. Tests and observations were performed on samples prepared in the laboratory and also removed from one cavity of a wall of a wood-frame house. The tests to characterize the foam included measurements of density, moisture content, shrinkage, and thermal conductivity, and analysis using X-ray diffraction, scanning electron microscopy, and thermogravimetry techniques.

It was found that the foam had a thermal conductivity comparable to that of other insulations used to retrofit walls of houses. Shrinkage could not be quantified, but was seen to be generally small. The moisture content of the foam removed from the house was about 2 percent. The results of the analytical measurements indicated that the laboratory-prepared samples and some of those removed from the house were not identical. It was suggested that further analysis be conducted to continue characterization of the foam.

Key Words: characterization; density; foam; insulation; magnesium oxychloride cement; moisture content; SEM; shrinkage; thermal analysis; thermal conductivity; X-ray diffraction

1. INTRODUCTION

1.1 Background

For the past decade, the Building Materials Division of the National Bureau of Standards (NBS) has provided technical assistance to the U.S. Department of Energy (DOE) in assessing the properties and performance of insulation materials. This technical assistance has included studies on new or non-conventional insulation materials used for, among other applications, retrofitting uninsulated cavities in walls of buildings. These studies have generally involved a preliminary phase that included publication of a review paper summarizing available technical data on the insulation, and recommending research to fill in gaps in the information data base. A notable example was a recent report on the properties and performance of urea-formaldehyde foam insulations [1]. A benefit in conducting such studies is the identification of research needed to enhance the performance of the nation's building inventory.

About 5 years ago, a new low-density (about 2.0 lbm/ft^3 or 32 kg/m^3) inorganic foam insulation became available in the U.S. [2]. The production of the insulation is based on magnesium oxychloride cement technology, but the chemical composition of the product has not been described in the archival literature. The manufacturer has indicated that the product is referred to in general terms as an oxychloride cement foam insulation, but is more properly described as a calcium magnesium oxychloride

silicate composition [2]. The insulation has been marketed for use in a variety of building constructions including new and existing wood-frame, steel-stud and masonry cavity walls, curtain walls, and for filling hollow-cores in masonry block walls.

The insulation is foamed-in-place from aqueous solutions at the building site in a manner similar to some plastic foams. The unset foam insulation may be pumped under pressure into closed cavities where access is obstructed and which may be difficult to fill with loose-fill insulation. After application, the foam hardens in place to a self-supporting material.

A review report on available data concerning the properties and performance of the oxychloride cement-based foam insulation was recently prepared [2]. The review indicated that data regarding the characterization of the foam as thermal insulation were not complete and that research was needed to provide data lacking on the performance properties of the foam. Properties for which the available data were limited included composition and structure, durability, effect on other building materials, shrinkage, and water absorption.

This paper presents the results of an initial investigation to obtain data on the properties and performance of the oxychloride cement-based foam insulation. Foam samples, prepared in the laboratory and removed from a wood-frame house, were included in

the study. Measurements of density, moisture content, thermal conductivity, and shrinkage, were combined with X-ray diffraction (XRD), scanning electron microscopy (SEM), and thermogravimetric (TG) analyses.

The analytical techniques (i.e., XRD, SEM, and TG) provide data concerning the chemical composition and cellular structure of the foam. Their use enables comparison of foam products from different installations to determine similarities and differences in composition, and whether compositional or microstructural changes are occurring in time.

1.2 Manufacture of Magnesium Oxychloride Cement-Based Foam

The manufacture of the low-density foam insulation has been previously described [2] and only a summary is presented here. Three major ingredients are involved: an aqueous cementitious slurry (based on magnesium oxychloride technology), a surfactant (or foaming agent) solution for cell generation, and compressed air. The equipment for generation of the foam on site includes a compressed air pump, and a mixing (or foaming) gun. The foaming agent is first pumped into the gun, where the compressed air mixes with it in an expansion chamber, and expands it into foam bubbles. Just before the bubbles exit from the nozzle of the gun, they are mixed with the cementitious slurry, which has been pumped through a separate line into the gun. The foam is then pumped from the gun. Expansion of the foam is complete at this

point and no expansion occurs after installation. Initial hardening of the foam proceeds to an extent sufficient for it to be self-supporting as it exits the gun. Complete hardening reportedly occurs a few weeks after application. When it exits from the nozzle of the foaming gun, the unset low-density foam reportedly contains about 50 to 60 percent water by mass [2].

2. EXPERIMENTAL

The following sequence constituted the experimental framework of the study. Details of each procedure are given in the subsections which follow. Laboratory foam samples were prepared in wooden boxes and, periodically, the boxes were opened to observe shrinkage. Foam specimens were removed from the opened boxes and the selected properties were determined. In addition, a section of a exterior wall of a house was opened to observe the foam in service and to obtain a specimen for laboratory testing.

2.1 Laboratory-Preparation of Foam Samples

A commercially available foam was prepared by manufacturer representatives according to their prescribed application techniques. The formulation of the foam was proprietary. Boxes, with interior dimensions of 610 x 610 x 90 mm (24 x 24 x 3.5 in.) and constructed from exterior-grade plywood and nominal 2x4 wooden studs, were filled with foam in the presence of NBS research personnel. After filling, the boxes were maintained with a long dimension in a vertical position at 23 $\pm$ 3 °C (73 $\pm$ 5 °F) and 45 $\pm$ 5 percent relative humidity. Periodically, the boxes were opened for shrinkage observations and removal of test specimens. These specimens are subsequently referred to as laboratory specimens.

2.2 Foam Samples from the Field

Foam specimens were obtained from a wood-frame house located in a rural northwest suburb of Washington, D.C. The wall construction consisted of nominal 2x4 wooden stud framing with a lath and plaster interior facing, and a rough-sawn wooden plank sheathing and painted wooden siding as the exterior facing. No membrane type vapor retarder was found in the wall cavity. The foam had been installed in the walls of the house about one year before the sampling. Specimens removed from the house are subsequently referred to as field specimens.

In October 1986, an exterior wall section, about 0.2 m^2 (2 ft^2) in area, was opened across one cavity (space between adjacent studs) to observe the in situ foam insulation and to remove samples. Some of the samples were sealed in glass jars for subsequent determination of density and moisture content. After removal of the foam, the empty cavity space was filled with fibrous glass insulation and the wall was closed using the original sheathing and siding.

2.3 Measurement of Linear Shrinkage and Density

Linear shrinkage of the foam samples in the wooden boxes was measured using a rule or calipers graduated in millimetres in accordance to a procedure that had been previously developed for cellular plastic insulation [3]. The boxes were opened for each measurement and then closed until the next determination.

Shrinkage measurements were made 28, 96, and 195 days after foaming.

For purposes of this report, "wet density" is the density at installation when the foam contains the water present during formation and application. Wet density was calculated from the mass of wet foam applied in tared 1-gallon metallic paint cans and the volume of the cans. Weighing was performed using an analytical balance with a sensitivity of 0.01 g.

To determine the amount of water present during application, the filled cans without lids were allowed to remain at ambient laboratory conditions and periodically reweighed. No provisions were made to avoid exposure to atmospheric carbon dioxide. Weighing was continued until a constant mass ($\pm$ 1 percent difference between consecutive weighings over at least 24 hours) was reached.

"Dry density" indicates the density of the foam insulation after it releases moisture present during application and it reaches equilibrium under normal laboratory environmental conditions. The foam in this condition is, thus, not totally "dry" in that no absorbed moisture is present.

The dry density of foam was determined using samples removed from the wooden boxes. The procedure was according to that described

in ASTM Test Method for Apparent Density of Rigid Cellular Plastics (D 1622). It was considered that the procedure in this standard was appropriate for the cementitious foam in the study, since some of its physical characteristics and density were comparable to that of many cellular plastics.

2.4 Moisture Content

Percent moisture content (MC) of the foam specimens was determined by measuring mass changes occurring during heating for 16 hours at 98 $\pm$ 2 °C (208 $\pm$ 4 °F) in a laboratory oven without mechanical convection:

$$MC\ \% = [(M_w - M_d)/M_d] \times 100$$

where M_w = mass of wet foam before heating, and
M_d = mass of dry foam after heating.

Mass of the specimens was determined using an analytical balance having a sensitivity of 0.01 g.

2.5 Measurement of Thermal Conductivity

Thermal conductivity measurements were carried out according to ASTM Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus (C 177). The metering area of the hot plate used was circular with a diameter of 400 mm (16 in.). The insulation specimen, having dimensions of 584 x 584 x 69 mm (23 x 23 x 2.7 in.) was placed in a wooden frame with a thin non-metallic bottom screen to hold the specimen during testing. The specimen was cut to the frame size with a wire to avoid damage. The thickness of

the specimen was slightly greater than the wooden frame so that the top surface of the specimen was in contact with the cold plate of the guarded hot plate. The bottom screen surface of the wooden frame was placed directly on the hot plate. Mean temperatures for the thermal conductivity measurements were 12, 24, and 38 °C (44, 75, and 100 °F), with a temperature differential across the specimen of 28 °C (50 °F) in all cases. The hot plate apparatus was operated in a one-sided mode, as described in ASTM Standard Practice for Using the Guarded-Hot-Plate Apparatus in the One-Sided Mode to Measure Steady-State Heat Flux and Thermal Transmission Properties (C 1044).

2.6 X-Ray Diffraction Analysis

X-ray diffraction (XRD) provides a means for identifying the crystalline phases in a sample [4]. X-rays are diffracted by planes of atoms in a crystal. Diffracted intensity at specific angles (2θ) is indicative of the presence of specific compounds in the sample.

Specimens were prepared for X-ray diffraction analysis by grinding samples of the foam insulation in an agate mortar and pestle. The resultant powders were back loaded into specimen holders. Back loading is a standard technique for preparing a powder sample for XRD analysis to minimize preferred orientation. Specimens were step scanned from 4° - 65° 2θ at a scan rate of 0.02°/second.

2.7 SEM Specimen Preparation and Analysis

Scanning electron microscopy (SEM) analyses were conducted to characterize the cellular structure of the foam specimens. The SEM technique provides good depth of field at high magnification [5]. In the present study, the foam specimens for SEM analysis were cut cylindrical in shape from larger samples using a cork borer. They had lengths of about 10 to 15 mm (0.4 to 0.6 in.) and diameters of 10 to 12 mm (0.4 to 0.5 in.). Two SEM specimen mounting stubs with 12 mm (0.5 in.) diameters were bonded to the ends of the cut specimens with an epoxy adhesive. When the adhesive had cured, the mounting stubs were pulled in opposite directions, breaking the single cut specimen into two mounted specimens with fractured surfaces.

The mounted specimens were sputter coated with a nominal 20 nm (8×10^{-7} in.) gold conductive film to prevent surface electron charging during SEM analysis. The fractured surfaces were examined in the SEM using an acceleration voltage of 30 kV and photographed at magnifications generally ranging from x20 to x2000.

2.8 Thermogravimetric Analysis

The thermogravimetry (TG) technique determines the weight change of a sample as a function of temperature [6]. Samples are normally heated at a selected rate, and the resultant thermograms (plots of mass vs temperature) provide data concerning the

thermal stability of the samples, for example, temperatures of thermal decompositions.

Foam specimens for thermogravimetric analysis were cut from large samples using a laboratory scalpel. The specimens (about 2.6 to 3.8 mg) were heated from 40 to 490 °C (104 to 914 °F) in nitrogen gas at a rate of 1 °C/min (1.8 °F/min). The nitrogen flow rate was 40 ml/min. The analyzer instrument was evacuated and then filled with nitrogen prior to each run.

3. RESULTS AND DISCUSSION OF THE LABORATORY MEASUREMENTS

3.1 Density and Moisture Content

3.1.1 Wet Density. The magnesium oxychloride based-foam insulation is applied wet. The moisture present during foam application must subsequently be dried from the wall to avoid damage to it. In the present study, it was of interest to determine the wet density of the foam and the amount of moisture released on drying, since these values can be related to field practice and the amount of moisture that a freshly-installed wet foam may release in the wall of a building.

Wet density was determined using metal cans to preclude moisture absorption by the container after filling with wet foam. The average wet density of the foam (three determinations) was 89 kg/m^3 (5.6 lbm/ft^3) with a coefficient of variation of 1 percent (Table 1). Thirty-eight (38) days were required for the foam in the metal cans to reach constant mass. During this period of time, the foam samples in the cans each lost about 135 g (0.30 lbm) of water or 40 percent of their initial wet mass. The dry density of the samples in the cans was not determined.

3.1.2 Dry Density. Dry density determinations were made on specimens cut and removed from the wooden boxes 28 days after foaming. The specimens removed from the boxes were found to contain moisture, indicating that the foams applied in the wooden boxes had not dried in 28 days. The specimen application boxes

were considered to be tight, resulting in slow drying. The foam specimens, removed from the boxes, were placed on an open shelf in the laboratory to dry. Nine days were required for the moisture to be lost from the specimens and equilibrium mass to be reached under normal laboratory conditions. During that time, about 25 percent of the initial mass was lost, which provided evidence that some of the initial moisture in the laboratory samples was lost in the wooden boxes.

The average dry density of the foam (4 determinations) was 44 kg/m^3 (2.8 lbm/ft^3) with a coefficient of variation of 4.4 percent (Table 1). When the specimens, on which dry density was determined, were heated for 16 hours at 98 $\pm$ 2 °C (208 $\pm$ 4 °F), they lost, on the average, an additional 1.7 percent of their mass. Heating for another 16 hour period produced no significant additional change in mass. The mass lost on heating was taken to be moisture.

Density specimens were again removed from the wooden boxes 96 days after foaming. In this case, the average value of dry density (4 replicates) was 46 kg/m^3 (2.9 lbm/ft^3) with a coefficient of variation of 3.5 percent (Table 1). In contrast to the specimens removed from the wooden boxes after 28 days, the 96-day specimens were essentially at equilibrium moisture content. Thus, it was considered that the foams in the wooden

boxes had completed drying within the 68-day period following the initial 28 days.

The dry density of three specimens removed from the house was also determined. The average value was 48 kg/m^3 (3.0 lbm/ft^3) with a coefficient of variation of 3.3 percent (Table 1). This density was considered comparable to the value for the laboratory specimens. The average mass loss on heating for the field specimens, taken to be moisture content, was 2.2 percent.

3.2 Shrinkage

Shrinkage of a foam insulation may cause air gaps that result in unwanted heat flow paths, and thus lower the thermal efficiency of the insulated wall [7,8]. When the present study was planned, it was intended to measure the percent shrinkage of samples prepared in the wooden boxes. However, the shrinkage measurements on the laboratory samples could not be quantified, but were found to be generally small (see the paragraphs that follow). Quantification of shrinkage was precluded because, where cracks attributed to shrinkage were present, they were narrow in width and randomly located across the bulk of the foam. Under such conditions, it was not possible to determine the accumulated gap space (necessary for a shrinkage calculation) created by the shrinkage cracks.

All wooden boxes were opened by removing a plywood face 28 days after filling. Three boxes for shrinkage were again opened after 96 and 195 days elapsed. The initial (28 day) observations showed that the boxes were essentially full. A few small gaps, penetrating through the foam from the front face of the box to the back face, were found in two foam samples. In all cases, the foam had adhered to the inner surfaces of the box. A narrow crack was visible near the box sides and near the perimeter of the foam samples. For each box, the width of the crack was immeasurably small using a rule or calipers graduated in millimetres. One box also had a narrow crack in the center of the foam. The presence of the cracks was attributed to minor shrinkage of the foam over the 28-day period. It was considered that the adhesion of the foam to the inner surface of the boxes was greater than the cohesive strength of the foam, resulting in the narrow shrinkage cracks near the perimeter of the foam samples, and not between the foam and the sides of the box.

After 96 and 195 days, when three boxes were again opened, the cracks were more readily apparent than during the first opening. In addition to the cracks near the perimeter of the foam, a number of cracks were also found randomly throughout the bulk of the foam. The width of the cracks were variable, with many being about 1 to 2 mm (0.04 to 0.08 in.) wide. The narrowest were considered immeasurably small, whereas the largest were found to be about 5 to 7 mm (0.2 to 0.3 in.) wide. It is noted that a

single gap of 7 mm (0.3 in.) over a distance of 610 mm (24 in.) would represent slightly greater than 1 percent shrinkage.

In outlining the scope of the study, it was not intended to measure foam shrinkage occurring in the metallic cans used for wet density determinations. However, after 6 months exposure in the laboratory, the foam samples in the cans were observed to have shrunk more than (4.5 to 6 percent) those in the wooden boxes. Although the significance of this observation, particularly as related to the use of foams in wood-frame construction, is not defined, this finding is reported since it raises concerns that the factors affecting foam shrinkage are not completely understood. In particular, one concern is whether foam shrinkage may vary depending upon the application environment and, perhaps, on the rate at which the freshly installed foam dries.

3.3 Thermal Conductivity

In planning the study, it was determined to perform thermal conductivity measurements on two specimens removed from the wooden boxes 28 days after foaming. These specimens would be allowed to remain at ambient laboratory conditions for one week before measurement. In this way, some residual moisture would remain in the foam, as would be considered typical of the foam in service where it would not be expected to be absolutely dry. The plan proposed to determine the thermal conductivity first on the "wet" specimens at a 24 °C (75 °F) mean temperature. Then, the

specimens would be dried in an oven and, subsequently, additional thermal conductivity measurements would be made over a range of mean temperatures. In practice, however, it was found that specimens large enough for thermal conductivity measurements were difficult to handle due to the inherent friability of the insulation. This precluded carrying out the proposed measurement procedure. The first specimen was damaged during drying in an oven after the initial thermal conductivity measurement at 24 °C (75 °F). Thus, upon removal of the second specimen from the application box, it was placed in the hot plate apparatus after remaining at ambient laboratory conditions for a week. Thermal conductivity measurements were made at three mean temperatures without oven drying of the specimen.

The results of the thermal conductivity measurements are given in Table 2. The values are comparable to those measured for other retrofit insulations such as mineral fiber or cellulose [9]. The foam specimens used for thermal conductivity measurement contained 2 or 3 narrow (< 1 mm) random cracks which formed while they remained at ambient temperature upon removal from the wooden boxes. The thermal conductivity values for the specimens with narrow cracks are reported, since cracks were observed in the foam sample examined in the house during the field phase of the study. The laboratory specimens for thermal conductivity were, thus, considered typical of that examined in the field. Also, as given in Table 2, the density of the laboratory specimens was

comparable to that of the foam removed from the house. The extent to which the cracks in the laboratory specimens contributed to unwanted heat flow during the thermal measurements was not determined, and requires further investigation.

As indicated in Table 2, thermal conductivity values for specimen 2 were measured for three mean temperatures. It is evident that these values increased as the mean temperature of the test increased. This relationship was linear with a correlation coefficient of 0.997. After completion of the thermal conductivity tests, a section of the specimen was dried in a laboratory oven. It lost 1.8 percent of its mass during heating, which was comparable to the mass loss found when other specimens in the present study were subjected to the conditions of drying.

The thermal conductivity value of 0.0430 W/m·°C (0.298 Btu·in./h·ft^2·°F), determined in the present study at 24 °C (75 °F) for a foam density of 47 kg/m^3 (2.9 lbm/ft^3), may be compared with a previously reported value. The literature reported value was 0.0371 W/m·°C (0.257 Btu·in./h·ft^2·°F) for a density of 33 kg/m^3 (2.1 lbm/ft^3) [2]. The literature reference did not specify the mean temperature of the test. The foam in the present study was found to have a thermal conductivity about 15 percent greater than that previously reported. The difference may possibly be attributed to the higher density of the foam in the present study, with perhaps some contribution due to the cracks in the

specimens. This, of course, assumes that the mean temperature of the literature test was about ambient laboratory temperature.

3.4 X-Ray Diffraction

X-ray diffraction (XRD) analyses were carried out on two laboratory specimens and two field specimens. One of the field specimens was taken from the vicinity of the outer side of the wall cavity, and the other was the vicinity of the inner side of the cavity.

Analysis of the X-ray diffraction patterns obtained from the two laboratory specimens indicated that the two were identical. The field specimen obtained from the outer side wall cavity gave a diffraction pattern which was also found to be virtually identical to those of the laboratory specimens. These three specimens were characterized by strong diffraction peaks at 2.37 (I/I_o*100)[1], 3.19 ($I/I_o \approx 60$), and 1.57 °A ($I/I_o \approx 50$). Cole and Demediuk [10] have published X-ray diffraction data for magnesium oxychloride cements including the 3-form, $3Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$, and 5-form, $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$. A comparison of the positions and relative intensities of the above peaks from the present study with the Cole and Demediuk data [10] indicated that the foams contained some magnesium oxychloride material, but the percentage was not ascertained. The comparison also did not provide determination as to whether the oxychloride present was the 3 form or the 5

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[1] I/I_o is the ratio of the given peak intensity to that of the most intense peak.

form, or if a mixture were present, the relative percentages of both.

Analysis of the field specimen from the vicinity of the inner side of the wall cavity also showed the presence of the above peaks. However, their intensities were significantly diminished in this case. Rather, in this instance the following strong peaks were observed: 2.99 (I/I_o*100), 3.19 (I/I_o*86), 11.5 (I/I_o*86), and 3.02 Å (I/I_o*79). The presence of strong 11.5 and 3.01 Å peaks is suggestive of the presence of the chlorocarbonate phase, $Mg(OH)_2 \cdot MgCl_2 \cdot 2MgCO_3 \cdot 6H_2O$, which forms as a result of the carbonation of the magnesium oxychloride cement. The reaction of carbon dioxide in air with magnesium oxychloride cements to form a carbonation product has been described [10,11]. Sorrell and Armstrong [11] have reported that the carbonation reaction can be beneficial and have a stabilizing effect on the magnesium oxychloride cements. It is noted that an investigation of possible carbonation of the foam insulation was beyond the scope of the present study, and should be the subject of further study.

3.5 Scanning Electron Microscopy

SEM analysis was conducted on two laboratory specimens. The results indicated that the microstructures of the two were generally the same. Fig. 1 shows a SEM photomicrograph taken of a laboratory specimen at x20 magnification. It is evident that the structure of the foam consists of cells that have a large variability in size. Each of the cells was seen to have a number

of thin windows and ribs connecting the windows. Some of the windows were open in that no membrane was present, whereas others had membranes in place.

Fig. 2 is a SEM photomicrograph at x200 magnification. Cell windows both with and without membranes are apparent. This figure also shows that the surface of the cells between the windows has a rough or pockmarked texture. In general, this surface roughness was typical of the cellular structure of the laboratory specimens. One exception was a limited area of one specimen in which a cell wall was found to have the appearance of needles.

SEM analyses were also performed on two field specimens for comparison with the photomicrographs of the laboratory specimens. As with X-ray diffraction, one field specimen was taken from the vicinity of the outer side of the wall cavity, and the other was taken near the inner side of the wall cavity. At low magnification (e.g., x20-x100), the microstructure of the field specimens appeared to be the same as that of the laboratory specimens. However, higher magnification observations (e.g., x500) of the surfaces of the cells indicated a marked difference between the two field specimens. The microstructure of the specimen from the outer side of the cavity was comparable to that of the laboratory specimens. In contrast, in the case of the field specimen taken from the inner side of the cavity, needles were observed on the

surface of many of the cells (Fig. 3).

The difference of surface characteristics of the microstructure of the two field specimens, observed by SEM analysis, was consistent with the results of the X-ray diffraction analysis. As discussed, X-ray analysis also showed the laboratory specimens to be essentially identical to the field specimen from the outer side of the cavity; whereas the field specimen from the inner side was different, possibly having formed some chlorocarbonate. The formation of a chlorocarbonate may produce a change in the microstructure that was observed in the SEM analysis.

It is of interest to determine whether the needles observed in the limited section of cell wall of one laboratory specimen was due to incipient formation of the chlorocarbonate or other reaction product. Further investigation is required to examine this question.

3.6 Thermogravimetric Analysis

Thermogravimetric (TG) analyses were performed on foam specimens prepared in the laboratory and obtained from the field. As was the case of the SEM analyses, the field foam specimens were taken from locations near the outer and inner sides of the wall cavity. The results are presented in Fig. 4, and summarized in Table 3. As evident from the Table 3, the mass loss results are given for three temperature ranges: a low range between 40 to

175 °C (104 to 347 °F), a medium range between 175 to 300 °C (347 to 572 °F), and a high range from 300 to 450 °C (572 to 914 °F). The ranges were selected so that comparisons could be made (see below) with data for magnesium oxychloride cements presented by Cole and Demediuk [10].

A notable feature in Fig. 4 is that the mass loss curves for the laboratory specimens and field specimens are not the same. In particular, the laboratory-prepared specimens underwent about 30 percent total mass loss over the heating range; whereas the field specimens lost about 40 percent of their mass. The increased mass loss for the field specimens is accounted for by greater mass losses in all three temperature ranges as compared to the laboratory specimens (Table 3). These mass loss results cannot, at this time, be related to the chemical composition of the foam, since its formulation is proprietary.

The TG evidence regarding relative mass loss for the three temperature ranges suggests that the composition of the laboratory specimens and both field specimens may be different. This finding contrasts sharply with the results of the X-ray diffraction and SEM analyses which indicated the field specimen

from the outer side of the cavity was essentially the same as the two laboratory specimens.

It is also evident from Fig. 4 that the mass loss curves for the replicate laboratory specimens are comparable. The shapes of the curves have marked similarity and the total mass loss over the heating range is closely the same (29 vs. 32 percent). This result supports those of the X-ray diffraction and SEM analyses which indicated that the replicate laboratory specimens were generally the same.

In agreement with the results of the X-ray diffraction and SEM analyses, the TG evidence also suggests that two field specimens may be different from each other. In particular, the curves in the low temperature range have different slopes. The total weight loss during heating for the outerside and innerside field specimens was 39 and 44 percent, respectively.

The results of the thermogravimetric analyses in the present study can be compared to those reported by Cole and Demediuk [10] for the 3-form and 5-form magnesium oxychloride cements. Their data (included Table 3) may be categorized into two major temperature regimes over which mass loss occurred: a low range from about 100 °C (212 °F) to about 175 °C (347 °F), and a high range of about 300 to 460 °C (572 to 860 °F).

In the low range, the magnesium oxychloride cements underwent dehydration to the same anhydrous phase [10]. The 3-form and 5-form lost about 34 and 27 percent of their mass, respectively when heated to about 175 °C (347 °F). These percents were essentially equal to the theoretical percentage of water of hydration present in each form. At higher temperatures, thermal degradation of the anhydrous phase occurred. The additional loss of mass by heating from about 300 to 460 °C (572 to 860 °F) was about 26 percent for both the 3-form and the 5-form.

A comparison of the TG results of the present study (Table 3) with those of Cole and Demediuk [10] suggests that the foam insulation is neither a pure 3-form or 5-form magnesium oxychloride cement, or a combination of the two. In particular, the percent loss in mass of the insulation specimens in the present study was considerably less, particularly over the low temperature range, than reported by Cole and Demediuk for the magnesium oxychlorides. Over the low temperature range, the mass loss in the present study was 16 percent or less (Table 3), whereas that from Cole and Demediuk was about 30 percent. The TG finding that the foam insulation is neither a pure 3-form or 5-form magnesium oxychloride cement is not considered surprising, since the insulation is prepared with fillers [2]. Fillers would be expected to alter the mass loss curves of the foam in relation to the pure magnesium oxychloride cements. Moreover, the manufacturer has indicated that the foam may be more aptly described as a calcium magnesium

oxychloride silicate composition [2]. Further research is needed to continue characterization of the foam insulation, and to resolve the apparent inconsistencies between the present X-ray diffraction and TG results.

4. RESULTS AND DISCUSSION OF FIELD OBSERVATIONS

The wall was opened from the exterior to provide access to the foam insulation. The oak plank sheathing had been installed diagonally to the studs, which did not allow opening more than one cavity without removing many pieces of siding board. Consequently, the observations of the in situ foam from the house were limited. It cannot be overemphasized that the observations reported here apply only to the house in question, because of their limited extent. The scope of this initial study of the properties and performance of these foams did not include a comprehensive field survey of a number of installations.

Upon removing the sheathing from the house, it was apparent that the installed foam had not completely filled the cavity space. The foam was in the center of the cavity, with unfilled cavity areas along both the left and right studs. The opening of an incompletely filled cavity filled was unfortunate, for it precluded taking measurements on shrinkage of the foam insulation. Shrinkage is normally estimated by determining the width of gaps created between the studs and the completely installed insulation.

The foam was found to be intact in the cavity. There was no evidence of crumbling, although the foam was friable to handle. Both horizontal and vertical cracks of varying widths were observed in the foam. Most cracks were estimated to be no greater than 1 to 2 mm (0.04 to 0.08 in.) in width. In some cases, the cracks were

considered immeasurably narrow, and in one case, the largest crack was found to be about 5 mm (0.2 in.) in width. Some of the cracks completely traversed the thickness of the insulation. Consequently, only relatively small pieces of the foam could be removed from the cavity. A sample large enough for a thermal conductivity measurement could not be obtained.

Four of the pieces of foam were weighed immediately in the field upon removal from the wall. They were again weighed periodically in the laboratory over a two week period during which they remained unsealed and exposed to normal room conditions. The samples showed no significant change in mass between the initial and final weighing, indicating that these foam samples removed from the wall contained no excessive accumulated moisture.

Upon removal of the foam from the cavity, a metal-sheathed electrical cable was found in the cavity mounted along the right stud. The foam had been in contact with the metal. No evidence of corrosion was visually observed. Finally, no indications of moisture problems (e.g., wet sheathing, water staining, and the like) in the cavity were seen. Measurements of moisture contents of the wooden members of the wall were not made.

5. SUMMARY AND RECOMMENDATIONS

5.1 Summary

This study was an initial investigation of the properties and performance of magnesium oxychloride-based foam thermal insulation. Tests and observations were performed on samples prepared in the laboratory and also removed from one cavity of a wall of a wood-frame house. The tests to characterize the foam included measurements of density, moisture content, shrinkage, and thermal conductivity, and analysis using X-ray diffraction, scanning electron microscopy, and thermogravimetric techniques. It is again emphasized that, because of the limited extent of the field observations and laboratory testing, the information reported applies only to the samples in the study. Based on the field observations and on the results of the laboratory tests, the following conclusions were made:

1. Thermal conductivity and dry density. The thermal conductivity of the foam was typical of that for other retrofit insulations ranging from 0.041 to 0.045 W/m·°C (0.28 to 0.31 Btu·in./h·ft^2·°F). Over the temperature range tested, the thermal conductivity increased linearly with an increase in the mean temperature of the specimen. The thermal conductivity was found to be 15 percent higher than that previously reported. This finding may have been due, in part, to testing specimens having dry densities that were 50 percent higher than those previously reported for the foam insulations, with some contribution due to minor cracks in the specimens. The density of the foam removed from the house

was comparable to that of the samples prepared in the laboratory for the present study.

2. Moisture Content. The foam insulation samples, prepared wet in wooden boxes in the laboratory, lost their initial moisture over 96 days while remaining under ambient laboratory conditions. The moisture content of the resultant foam was about 2 percent. This value was comparable to that of the 1 year-old foam removed from the wood-frame house, indicating that, in this case, the insulation installed wet in the wall had lost the water present during application.

3. Shrinkage. Shrinkage of the foam insulation was observed for the laboratory samples prepared in wooden boxes, as indicated by cracking of the foam. The shrinkage was generally small, as evidenced by the widths of the shrinkage cracks. However, the extent of shrinkage could not be quantified. Because the shrinkage cracks were narrow and randomly located across the bulk of the foam, it was not possible to determine the accumulated gap space necessary for a shrinkage calculation. In the case of the field sample, incomplete filling of the cavity precluded obtaining shrinkage measurements.

4. Field Observations (Other Than Shrinkage). The observations from the field indicated that, for the cavity opened for inspection, the installation of the foam was incomplete. The installed foam was intact in the cavity without evidence of crumbling. Nevertheless the foam was friable to handle. The field observations also indicated no visible evidence of moisture problems in the cavity

examined.

5. Analytical Techniques. X-ray diffraction, scanning electron microscopy, and thermogravimetric analysis offer useful techniques for characterizing the magnesium oxychloride-based foam insulations. The X-ray diffraction analysis provided evidence that magnesium oxychloride cement was present in the insulation, but did not indicate whether it was the 3-form, 5-form, or a combination of both. X-ray diffraction indicated that the laboratory sample and the field sample from the outer side of the cavity appeared to be identical. The technique also suggested that the field sample from the inner side of the cavity may have contained some chlorocarbonate, which may have formed through the reaction of the magnesium oxychloride with the carbon dioxide in air. The SEM analysis was consistent with the X-ray diffraction analysis in that the laboratory samples, in general, had microstructures similar to those of the field sample removed from the outer side of the cavity. Conversely, the microstructure of the field sample removed from the inner side of the cavity was different, indicating that some change may have occurred. The results of the thermogravimetric analysis were not completely consistent with the those of the other two analytical techniques. The thermograms for the two laboratory samples were comparable with each other. However, the thermograms for the laboratory samples were considered different from those of both field samples. In addition, the thermogravimetric analysis results were not consistent with thermograms published for pure magnesium

oxychloride cements. This latter finding was not surprising, since it has been reported that the foam is prepared based on magnesium oxychloride technology, but contains fillers and other ingredients which may affect its composition.

5.2 Recommendations

As indicated, this initial study was limited in scope. Moreover, the data obtained on foam characterization using the various analytical techniques were, in some cases, inconsistent from one method to the next, pointing out the need for additional characterization. Thus, because of the limited scope of the study and inconsistencies in some of the analytical data, recommendations are made that the X-ray diffraction, SEM, and thermogravimetric analyses be extended to provide further characterization of the foam. Investigations should include studies to quantify the composition of the cement-based insulation, and to evaluate the potential of the cement to undergo carbonation and the effect of carbonation on insulation performance.

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Table 1. Results of Density Measurements

Specimen	Determinations No.	Density kg/m^3	Density lbm/ft^3	COV[a] %
Lab, wet	3	89	5.6	1.0
Lab, dry (28 days)	4	44	2.8	4.4
Lab, dry (96 days)	4	46	2.9	3.5
House, dry	3	48	3.0	3.3

a. coefficient of variation

Table 2. Results of the Thermal Conductivity Measurements.

Specimen No.	Density[a] kg/m^3 (lbm/ft^3)	Mean Temp. °C (°F)	Thermal Conductivity W/m °C (Btu in./h ft^2 °F)
1	47 (2.9)	24 (75)	0.0430 (0.298)
2	46 (2.9)	12 (44)	0.0408 (0.283)
2	46 (2.9)	24 (75)	0.0425 (0.294)
2	46 (2.9)	38 (100)	0.0450 (0.312)

a. Density was determined after the thermal conductivity was measured.

Table 3. Results of Thermogravimetric Analyses.

Specimen No.	Temperature Range		
	Low	Medium	High
	40 - 175 °C (104 - 347 °F)	175 - 300 °C (347 - 572 °F)	300 - 450 °C (572 - 842 °F)
	Mass Loss, %	Mass Loss, %	Mass Loss, %
Lab. 1	11	2	16
Lab. 2	11	2	19
Field Outer[a]	15	3	21
Field Inner[b]	16	4	24
3-form MgOCl[c]	34	negligible	26
5-form MgOCl[c]	27	negligible	26

a. Specimen was taken from the vicinity of the outer side of the cavity.
b. Specimen was taken from the vicinity of the inner side of the cavity.
c. Data are taken from Cole and Demediuk [10].

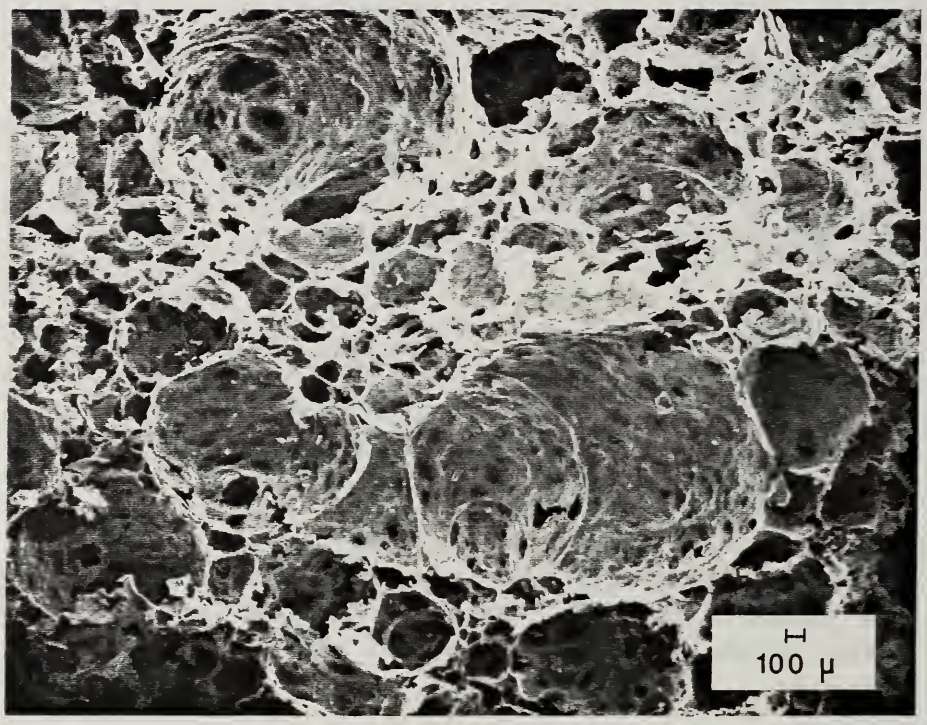

Fig. 1. SEM photomicrograph of a laboratory foam specimen at x20 magnification.

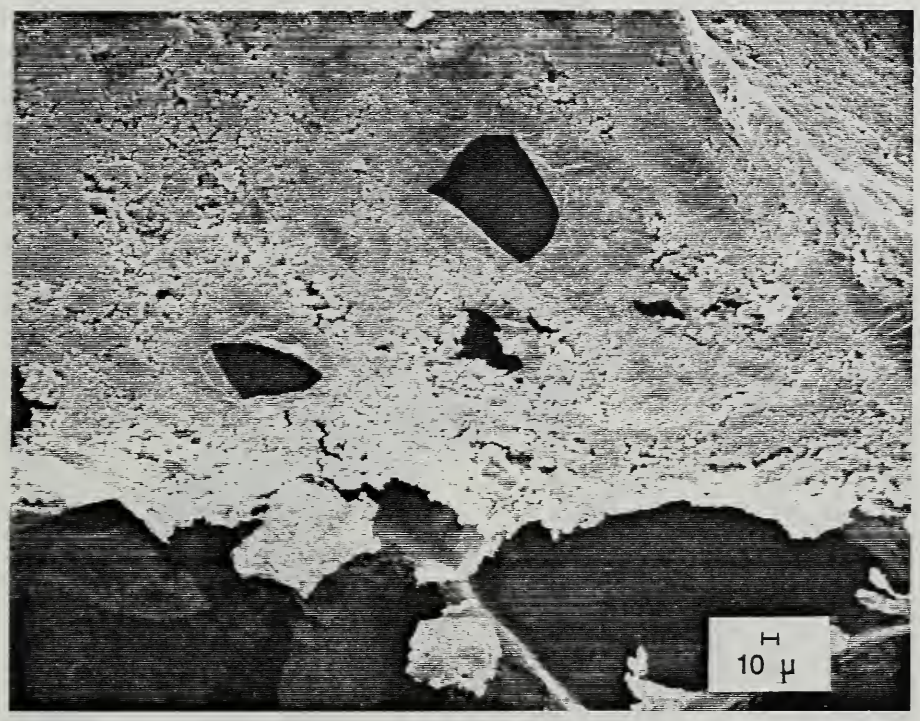

Fig. 2. SEM photomicrograph of a laboratory foam specimen at x200 magnification.

Fig. 3. SEM photomicrograph of a field foam specimen at x2000 magnification. The specimen was removed from the inner side of the wall cavity.

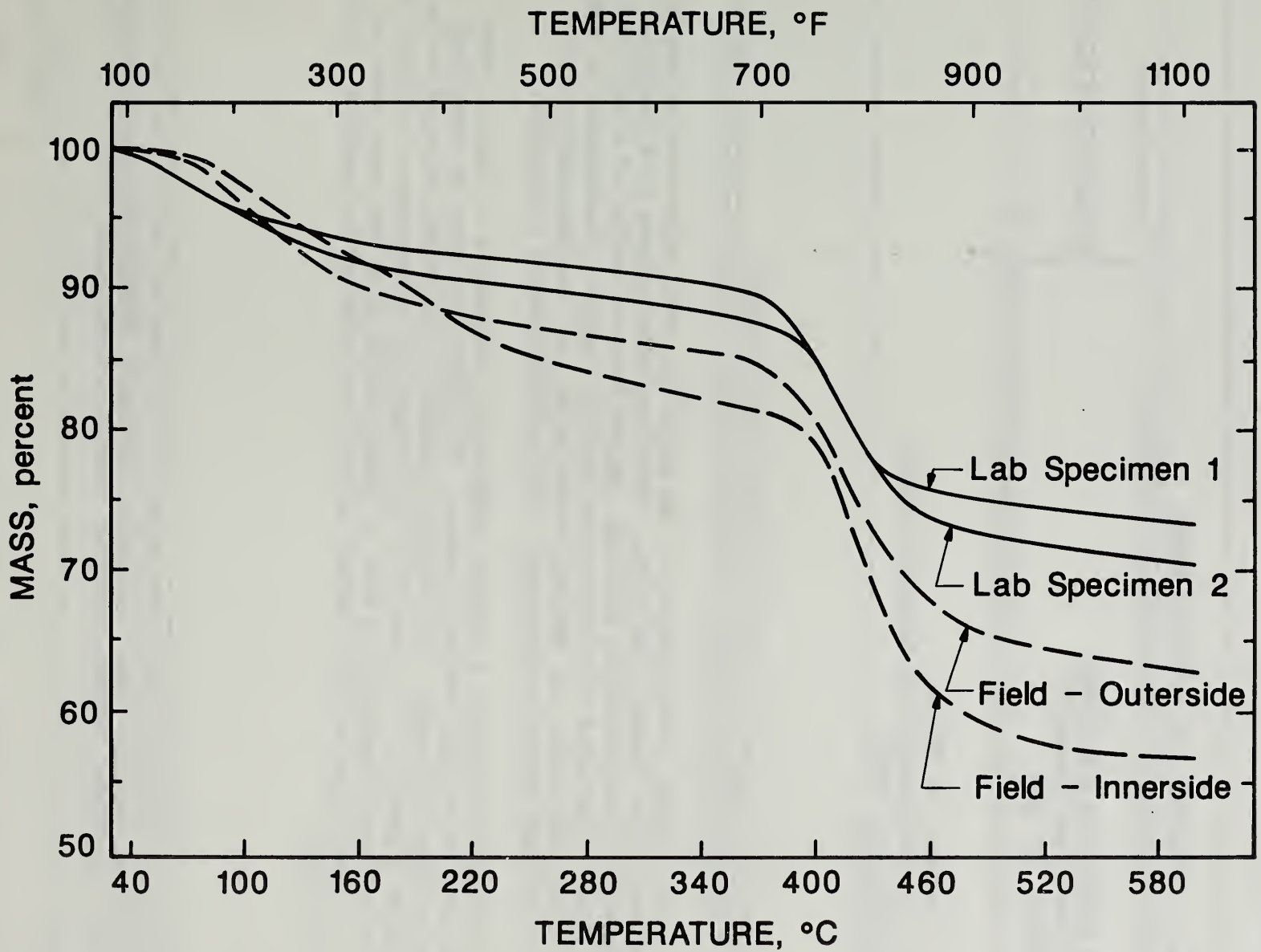

Figure 4. Results of the TG analysis. Specimens were heated in nitrogen gas at 1°C/min (1.8°F/min).

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This study is an initial investigation of the properties and performance of magnesium oxychloride-based foam thermal insulation. Tests and observations were performed on samples prepared in the laboratory and also removed from one cavity of a wall of a wood-frame house. The tests to characterize the foam included measurements of density, moisture content, shrinkage, and thermal conductivity, and analysis using X-ray diffraction, scanning electron microscopy, and thermogravimetry techniques.

It was found that the foam had a thermal conductivity comparable to that of other insulations used to retrofit walls of houses. Shrinkage could not be quantified, but was seen to be generally small. The moisture content of the foam removed from the house was about 2 percent. The results of the analytical measurements indicated that the laboratory-prepared samples and some of those removed from the house were not identical. It was suggested that further analysis be conducted to continue characterization of the foam.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

characterization; density; foam; insulation; magnesium oxychloride cement; moisture content; SEM; shrinkage; thermal analysis; thermal conductivity; X-ray diffraction

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