

REFERENCES

1. Drywall Research Committee: Drywall construction manual. 21-24, The Building Center of Japan, 1992.
2. The Japan Building Disasters Prevention Society: Investigation of Fire Performance of Drywall Partition on Electric Appliances, 21-33, 120-135, 1999.
3. Japan Gypsum board Association: Gypsum Wallboard Handbook, 29-47, Japan Gypsum Association, 1998.
4. Japanese Standard Association: JIS Handbook (Building II), Thermal insulation of artificial mineral fibre, A 9504, 964-967, 1995.
5. ISO/TC2: FDIS 834-1, Fire resistance tests - elements of building construction-part 1: general requirements, 7, ISO Central Secretariat, 1998.
6. M.Iiji: Specification of tapered edge drywall, Rep. of the 3rd meeting of fire performance evaluation, 1998.
7. A.Iida, et al.: Evaluation of fire resistivity of drywall after hose stream test, 89-90, Trans. of annual meeting of AIJ, Hiroshima, 1999.
8. S.Sugahara: A thought on charring characteristics of timber, 167-175, Disaster research 10, 1987.

Morphological Research on Gypsum Board Exposed to Fire Based on SEM and XRD Analysis

SHINICHI SUGAHARA, AKIHIKO IIDA and MASAMICHI TAMURA

Department of Architecture, Graduate School of Engineering

University of Tokyo

7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656, Japan

ABSTRACT

Gypsum board is used, in general, as interior finish material in building, aimed at functional efficiency, i.e.: fire resistance, insulation, sound barrier and so on. However, there is little research on substantial transformation of gypsum board under fire exposure necessary to settle thermal properties such as coefficient of thermal conductivity, heat transfer expansion, and specific heat [1]. In this study, fire-tested specimens were analyzed by using scanning electron microscope (SEM) and X-ray diffraction (XRD) in order to grasp the relationship between these substance transformation and fire performance test. Test data obtained from DTA and TG test, XRD and SEM observation showed the transformation of bassanite or anhydrite around 135 °C could be effective for suppressing temperature-rise in drywall.

KEYWORDS - *gypsum board, XRD, SEM, fire-resistance test, interior finishing materials, bassanite, anhydrite*

INTRODUCTION

Interior finish materials would be selected rationally if fire protective performance could be decided in accordance with fire scenario including thermal properties of gypsum board. In a fire, gypsum wall or ceiling board dehydrates by 1.5 molecules of H₂O and continuously transforms into CaSO₄·0.5H₂O at about 100 °C. Gypsum is ultimately transformed to CaSO₄, such as polymorph of anhydrite [2]. This study is aimed at clarifying substantial transformations in a fire by analyzing SEM, X-ray, DTA and TG tests.

EXPERIMENTALS

SAMPLES AND HEATING PROCEDURE

Gypsum boards prescribed in JIS A 6901 and JIS A 5430 were used in this study [3, 4], whose size were 100 × 100 × 15 mm. At first, electric furnace was continuously heated up to 300 °C and then the sample was quickly set into the furnace. Each sample (Specimen No. 8 – 14) was collected when its temperature reached to a stable point, i.e.: 90, 120, 130, 135, 170, 180 and 385 °C.

Secondly, other samples (Specimen No. 1 – 7) were collected in every 100 °C interval. All heated samples were cooled down and stocked in dry desiccator. Samples (Specimen No. 15– 18), as shown in Table 3 and Figure 1, were taken out from gypsum board after 2-hour bench scale fire test of the ISO834 [5].

Several samples were served to SEM observation. And powdered samples were used for DTA, TG and X-ray diffraction test, whose devices and measurement conditions are explained as follows.

Table 1: specimens of No.1-7 exposed to stable temperature rate

Specimen No.	Furnace Temperature (°C)	Time to stable Temp. (min)	Identified substance
1	200	80	anhydrite, bassanite
2	300	60	anhydrite, bassanite
3	400	50	anhydrite
4	500	40	anhydrite
5	600	30	anhydrite
6	700	30	anhydrite
7	800	21	anhydrite

Table 2: specimens of No.8-14, exposed to 300 °C

Specimen No.	Time to final temp. (min)	Final Temperature (°C)	Identified substance
8	2	90	gypsum, bassanite
9	4	120	gypsum, bassanite
10	10	130	gypsum, bassanite
11	19	135	bassanite
12	22	170	bassanite
13	26	180	bassanite
14	35	385	anhydrite, bassanite

Table 3: Samples exposed to the time-temperature curve of ISO834

specimen No.	contents
15	before heating
16	exposed side
17	about 5 mm in depth from exposed surface
18	exposed side

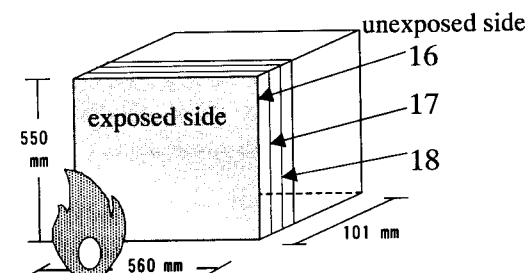


Figure 1: An illustration of specimen exposed to standard fire test (one side heating) and each number shows sampling layer

X-ray powder diffraction

X-ray diffractometer, RIGAKU Ultima RINT 2100 (XRD) was used. Data was corrected every after measurement of silicon standard sample. Goniometer was settled as a scanning speed of 4.0 degree/min and range 3.0-80.0 degree. X-ray was generated with a target of Cu at 50 kV, 40 mA and

passing through divergence slit at 1°, scatter slit 1° and receiving slit 0.3mm.

Differential thermal analysis (DTA)

DTA, RIGAKU TG-DTA thermoplus TG8120 was operated. This device was used for measuring weight difference according to the thermal changes based on standard specimen (Al_2O_3). The heating rate of 20 mg sample was 10 °C/min on the platinum pan [6]. The thermogravimetry analysis was also carried out from which the thermal kinetic change was obtained

Observation by scanning electron microscope (SEM)

Gypsum board samples were mounted on the specimen stage spattered with gold under vacuum environment. Scanning electron microscope, JOEL JSM-T100 was operated at 25 kV with 50-10000 magnification. Energy dispersive X-ray spectrometer of KEVEX7000 attached in SEM was used for elemental analysis of gypsum and aggregation materials.

RESULTS AND DISCUSSIONS

Temperature distribution at the central part of gypsum board is shown in Figure 2 when furnace temperature inside was kept at 300 °C. Rapid temperature-rise after heating becomes slow at around 90 °C and next slowness occurs at about 120 °C and then the first equilibrium condition begins around 135 °C. After that, temperature rises again and temporarily exceeds furnace temperature. Until finishing second equilibrium condition, weight loss decreased gently up to 20 percent. The heat flow rate by DTA test (Figure 5) and weight loss percent of H_2O , 1.5 molecules were almost in the same manner.

According to the results of the identification by XRD analysis (Figure 3), bassanite begins to form at about 80 °C. There is the coexistence of bassanite and gypsum until gypsum disappears at about 135 °C. During furnace atmosphere is kept at around 135 °C, anhydrite coexists with bassanite, and around 400 °C the components gradually change to anhydrite only. Cooling process in desiccator has little influence to substance transformation, which is cleared by the X-ray analysis. Although specimens were transformed to anhydrite, a little bassanite were observed in the sample ambient to unexposed side (about 230 °C). Average temperature reaches to 1070 °C in furnace heated for two hours along with ISO time-temperature curve. DTA curve (Figure 5) showed two peaks at both 130 °C and 145 °C, which indicates composite transformation. Specimen 15 of unheated gypsum board includes a large amount of residues filled with curing water at spherical spacing range of 30-40 μm .

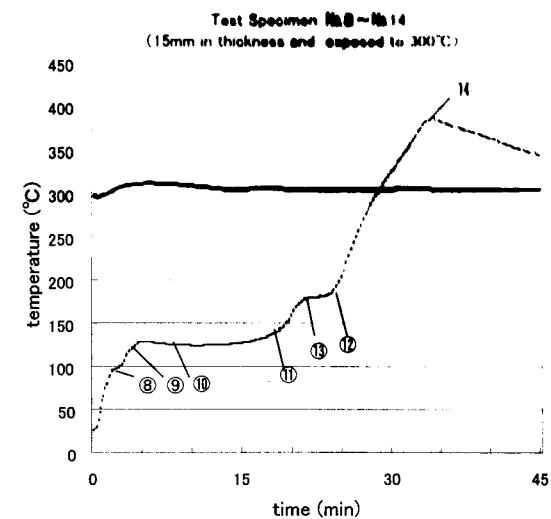


Figure 2: Temperature rise of test specimen. Solid line: furnace temperature. Dash line: specimen temperature. Number in round: sampling point of test specimen as shown in Table 2. The thermal rise over 300 °C (furnace temperature) had two characteristics, i.e. the thermal peak and the shift to furnace temperature (solid line). Although there exists geometrical furnace features and chemical transformation from III to II-type of calcium sulfate in the range of 230°C - 350 °C, additional discussions would be required for these high temperature areas.

The two shapes of prismatic and platy were formed in gypsum board specimen. The amount of former substance were more than that of the latter. The prismatic crystals show approximately 5 μm in length and 0.5-1.0 μm in width, as if platy shape would be filled with them (Photo 1B). The platy crystals show about 0.5-10 μm in thickness and more than 1.0 μm in width. In some cases, they were recognized in aggregation of crystals consisting of several prismatic shapes. All the crystal planes of this specimen had not remarkable cracks and were clearly flat. Each crystal particle exists in disordered directions and the gap among crystals was spacious. Additionally, glassfibers of about 10 μm in width were observed to be in disordered direction.

The test specimen, as shown in Table 1, indicates the growth of cracks often grown (Photo 1A: especially in specimen 6). Cracks of specimen occur in parallel to the elongation, which often grew to deformation or break. The part observed as wrinkle is evident to be composed of small cracks, associated with curving and breaking. This is especially remarkable in prismatic crystal. Large cracks with disordered direction were relatively recognized on the surface of platy-shaped crystals.

These phenomena grew along with temperature rise. In many cases, a great deal of wrinkles and cracks on the whole of the crystal plane developed apparently to broken parts throughout separation of crystal.

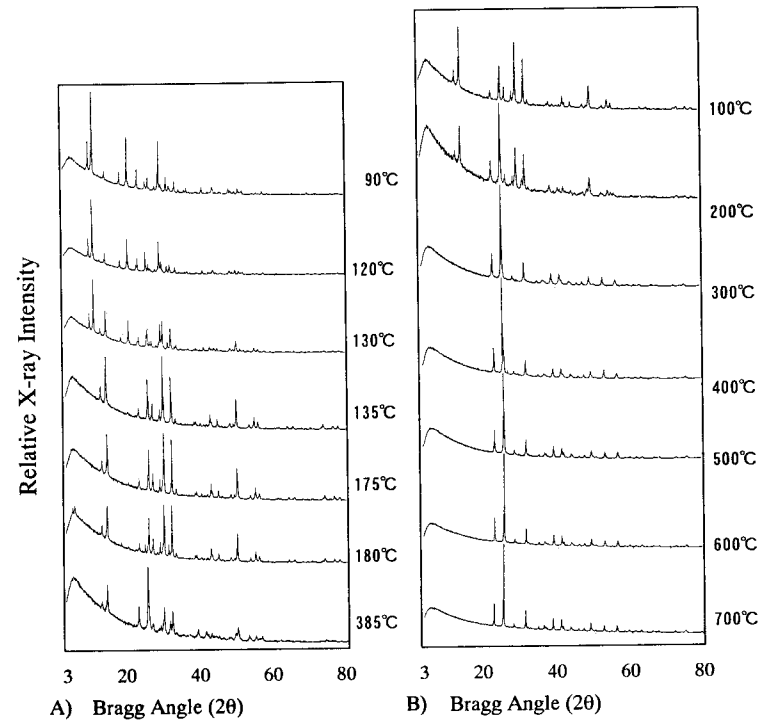


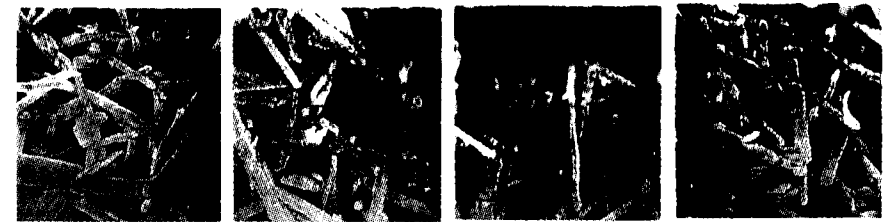
Figure 3 X-ray diffraction patterns of dehydrated test specimens at resulted in.

A) Each temperature in chart shows seven characteristic points.

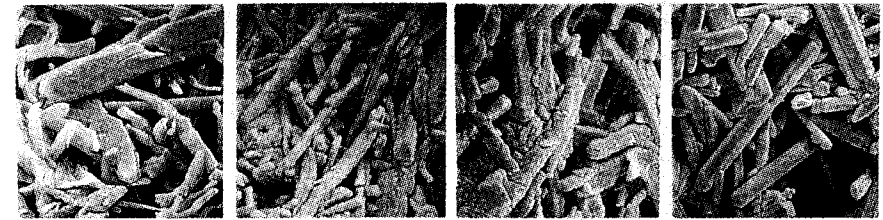
B) Furnace was heated by every 100°C up to 800°C.

In the case of test specimens in Table 2, the above-mentioned description were confirmed by the fact that wrinkles or cracks were limited to the surface of crystals exposed to higher temperature than that of specimen 2 heated for 80 minutes at 200 °C. As a result, these samples have similar crystal shapes.

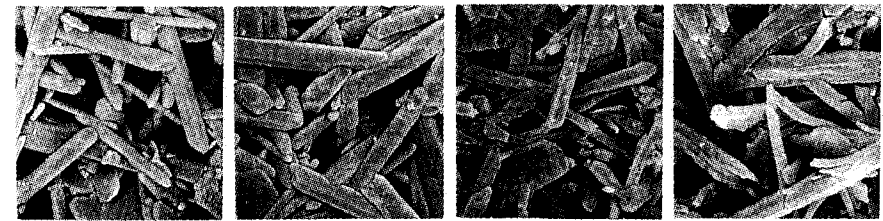
An interesting result could be obtained from the observation of test specimen served to fire resistance test. The heated side of the test specimen, showed a large amount of small particles, about



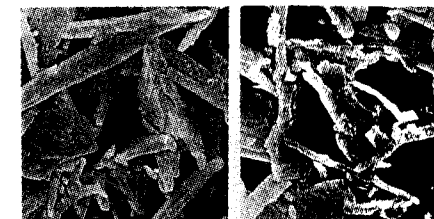
Specimen 1 Specimen 2 Specimen 3 Specimen 4



Specimen 5 Specimen 6 Specimen 7 Specimen 8



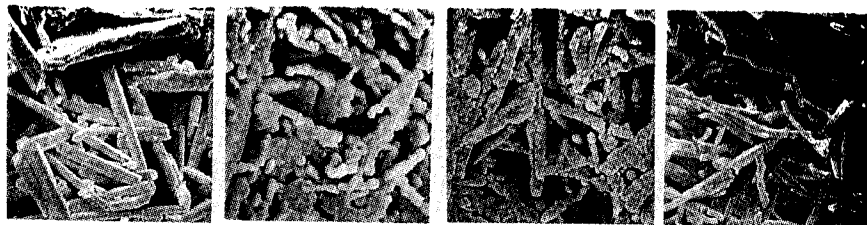
Specimen 9 Specimen 10 Specimen 11 Specimen 12



Specimen 13 Specimen 14

5 μm

Photo 1A : The representative images of SEM observed gypsum board specimens. They were under each above-mentioned condition, corresponding to each table.



Specimen 15 Specimen 16 Specimen 17 Specimen 18

Photo 1B: The representative images of SEM observed. Specimen 15 unheated, Specimen 16-17 by 2-hour-fire resistance test.

5 μ m

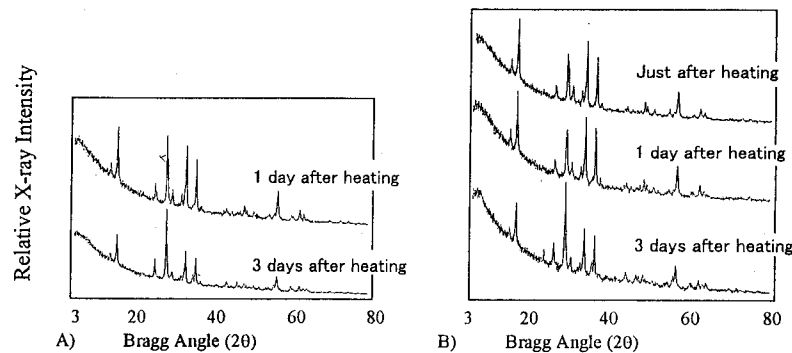


Figure 4: X-ray diffraction patterns: (a) of samples in a room and (b) of the samples reserved in desiccator.

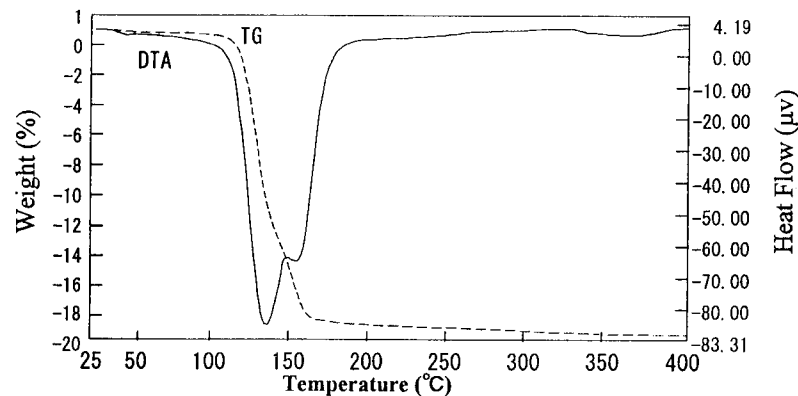


Figure 5: The gypsum spectra of DTA (solid line) and TG (dashed line)

1.0 μ m in size, which developed on the surface of each crystal. Although the temperature rise of specimen 17 were not measured, similar changes were recognized when heated for 21 minutes at 800 $^{\circ}$ C. The back of underlayered sheet of specimen 18 presented a similar feature to specimen 2-4. The upper sheet of this specimen became so fragile that it was easily rubbed by hand to disentangle.

CONCLUSION

As a result of this study, it is suggested that if heating would be limited under 200 $^{\circ}$ C thermal path in cooled-down gypsum board could be estimated from the occurrence of bassanite and extinction of gypsum. On the other hand, it would be difficult to deduce temperature rise history upper than 200 $^{\circ}$ C. The first reason is considered difficult to determine thermal path due to reverse reaction in cooled-down gypsum with associated minerals. The second one is due to coexistence of several phases of α - CaSO_4 and the other associated minerals such as bassanite and anhydrite. Peak resemblances method would be hard to determine characteristics of composed substances in more detail, in spite of the various associated phases. However, thermal path in gypsum board could be estimated to a certain extent, because of the coexistence of the mineral phases imposed within the order in Table 2. But it is essential for another additional examination of thermal path to be put in practice for clarifying the possibility of identifying reverse substances. Examination on long-term alteration of gypsum could clear whether efficiency of water could be ignored or not. Moreover, this study should be executed under additional condition including hose stream test, as shown in JIS A 1304 or ASTM E 119 [1, 7, 8, 9]. In the same way, examination with XRD is necessary to chase the morphological changes by analyzing under heating and cooling process.

Decrease in the volume of gypsum at about 100 $^{\circ}$ C is resulted from release of dehydrated water by, 2/3 molecules of H_2O along with transformation of crystal structure, effective for fire resistive performance. Subsequently, crack or wrinkle appears in the crystal because dehydration of half molecules of H_2O , id est; transformation from $\text{CaSO}_4 \cdot 1/2\text{H}_2\text{O}$ to CaSO_4 occurs around 250 $^{\circ}$ C. In addition, it should be indispensable to examine round shape covered with particle-like materials emerged around 800 $^{\circ}$ C. According to this observation, morphological changes could be characterized on the crystal structure exposed to fire furnace. Another result, which II-type of CaSO_4 was not hydrated under wet condition in a week just after heating, suggests that water damages by sprinkler and hose stream cannot be almost excluded under high temperature condition at least more than 400 $^{\circ}$ C. On the other hand, change of temperature around from 400 $^{\circ}$ C to room temperature may affect rapid transformation of several phases of $\text{CaSO}_4 \cdot x\text{H}_2\text{O}$. Thus, low temperature property of gypsum board could be clear in detail if analysis of water content would be possible just after heating. Accumulation of additional data on the crystal morphology could be able

ACKNOWLEDGEMENTS

We thank Mr. T. Nishiwaki, Central Research Institute, Asano Co. Ltd. for their help during X-ray diffraction test, with expert guidance and critically reviewing the manuscript, Mr. M. Iiji, Executive Manager, Japan Gypsum Association and Mr. J. Takase, Fire Officer, Tokyo Fire Department, for their contribution to this research, and also thank Mr. S. Otsuka and Mr. Y. Suzuki, Graduated School of Engineering, Univ. of Tokyo, for their help in operating electron microscope and in operating X-ray diffractometer, respectively

REFERENCES

- [1] J. Mchafley *et al.*: "A Model for predicting heat transfer exposed to fire", p. 297-305, Vol. 18, Fire and Materials (1994)
- [2] Hurlbut *et al.*: ed. "Manual of Mineralogy", Wiley, pp. 351-353 (1985)
- [3] JISC Committee: JIS A 6901, "Gypsum boards", JSA (1997)
- [4] JISC Committee: JIS A 5430, "Fiber-reinforced cement boards", JSA (1995)
- [5] ISO/FDIS 834-1: "Fire resistance tests - elements of building construction" (1997)
- [6] Izumi *et al.* (eds): "the 2nd edition of analytical devices guide (in Japanese), Vol.3, Kagakudoujin, pp. 1-18 (1996)
- [7] JISC Committee: JIS A 1304, "Method of fire resistance test for structural parts of buildings", JSA (1994)
- [8] ASTM Committee E-5: ASTM E 119-88: Annual book of ASTM standards, Vol. 04.07, pp. 412-432 (1994)
- [9] A. Iida *et al.*: Evaluation of fire resistivity of drywall after hose stream test "(in Japanese), p. 89-90, trans. of annual meeting of AIJ, Hiroshima (1999)
- [10] T. Harmathy, DBR Paper No. 1080, National Research Council of Canada, Ottawa (1983)
- [11] U. Schneider: Behavior of concrete at high temperatures (1982)

An Extrapolation Method of Steel Column Temperature Rise under Fire Resistance Tests by Using Parameter Estimation Technique

AKIKO NANBU and TAKAO WAKAMATSU
Department of Architecture, Science University of Tokyo
Yamasaki 2641, Noda, Chiba 278-8510, Japan

KAZUNORI HARADA
Department of Architecture and Environmental Design, Kyoto University
Yoshida-honmachi, Sakyo, Kyoto 606-8501, Japan

SHUITSU YUSA
Building Research Institute, Ministry of Construction
Tatehara 1, Tsukuba, 305-0806, Japan

ABSTRACT

To derive design diagrams of insulated steel columns, a methodology was proposed to extrapolate the temperature rise of specific steel column assembly under fire resistance test to those differing in cross sectional area of steel. Having a set of steel column temperature measurement, the thermal conductance between fire resistance furnace and steel was estimated by the technique of parameter estimation. Then, using the estimated thermal conductance, temperature rise of steel columns with arbitrary cross sectional area was calculated while keeping the construction of insulation unchanged. The results were compared with fire resistance test results that were conducted separately. The agreement was good or conservative if the cross sectional area were larger than the reference cross sectional shape. Thus it was shown that the methodology is feasible to develop design diagrams for insulated steel columns. A practical chart and simple design formula was produced for columns insulated by calcium silicate boards.

Key words: fire resistance tests, steel columns, fire resistance insulation, parameter estimation, extrapolation, design diagram

INTRODUCTION

To prevent the structural frame from collapse during fire, fire resistance is essential to the members of structural frame. In case of steel frame, mechanical properties such as elastic modulus and the yield strength are decreased as the steel temperature rises, which results in