Comparison of The thermal Characterization of Refractory board for Concrete consist of Flame Retardants and Compound Material

Il Young, Jang., Woo Jung, Jung., Se A, Cho

Abstract—The concrete structures might reach up to the highest inner temperature 1,400 °C of the structures when there is a fire. At this time, there could be spalling phenomenon as a feature of concrete and fatal damages like falling off in structure stability and durability because of the temperature increase in main reinforced bar. In the research, by using fireproof board containing silicon compound as a base with flame retardants has been tried to verify the possibilities of securing the structural stability, durability, and fire resistance. To make it possible, analysis on thermal feature and the performance of fire resistant were carried out with manufactured specimens which is silicon compounds produced through the changes of weight ratio of 25~40wt% with silicon compounds like ATH, MDH, and MC. As the result, all the specimens were separately verified as to its excellence for fire resistance and the possibility that could make stability, durability, and performance of fire resistance secured as a fireproof board was also verified when they are applied to concrete structures.

Keywords—silicon rubber, fire resistance, flame retardant, Flame Test, thermo gravimetric analysis (TGA) Limiting oxygen index (LOI)

1. Introduction

In modern society, the structures appear in the form of high rising buildings and bigger size due to advanced industrialization. In addition to the development of traffic and technology, they are threatened by the various outbreaks of fire such as the increase in traffic volume, electricity, gas usage and etc. These conditions mean that the chance of outbreak of big fires gets higher, especially in case of the skyscrapers and long tunnels, in the event of fire, it's hard for them to be put out in early stages and so it may result in the increase in big human or material loss because of the inner

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heat and toxic gases. In case of most concrete structures, high temperature heat cause the occurrence of explosive fractures and the rise of the temperature in the main reinforced bar reduce the durability of the subsidiary materials remarkably. Consequently, it brings the fatal cause for lowering the structural safety of the whole structure. The concretes are generally excellent in fire resistance However, general solidity, explosive fractures, and the low efficiency of fire resistance have recently become an issue as the use of high performance concrete grows. In order to control the rise of the temperature on the surface of the concretes, the application of the original fireproof board has problems like occurrence of harmful gases, deficiency in fireproof performance, and high expenses in the event of fire. The research discusses the fire resistant board which can hold improved fireproof performance through the effective control on the rise of the temperature. With regard to the domestic regulations on the fireproof performance, 'Management Regulations on the fireproof performance of the high solid concrete columns and beams(Ministry of Land, Transport and Maritime Affairs, 2008)', 'Acknowledgment and Management standards of Fireproof Structure(Ministry of Land, Transport and Maritime Affairs, 2012), 'Standards for Fire retardant performance and prevention structure to fire spreading of finishing materials for structure(Ministry of Land, Transport and Maritime Affairs, 2012) were referenced. After producing testing materials based on silicon, checking feature analysis of the heat (TGA), analysis of fire retardant (LOI), and analysis of fire resistance (Flame Test) were performed.

II. Methods of Study

A. Fireproof materials

The silicon compound used in this research which is two liquid typed and hardens in normal temperature consists of part A, Methylhydrosilane and part B, Hydrosilane. They are hardened after combination at a one to one ratio. Part A and B are a principal member and a hardener respectively. They are hardened by the bridging reaction when they are mixed.

As added fireproofs for improving fire resistant performance to the base of silicon compound are the inorganic flame retardants; ATH (Al(OH)3), MDH(Mg(OH)2), and nitrogenous flame retardant; MC(Melamine Cyanurate).



B. The production of specimen and experiment

The fireproof board was made from silicon compound and flame retardant mixed at the weight ratio under the table 2.1. After that, it was poured into a mold sized 400 X 100 X 20mm and then completely hardened in the preheated drying furnace at 80 $^{\circ}$ C for 24~48 hours.

TABLE I. COMPOUNDING RATIO FOR SILICON/ FLAME RETARDANT

Section	Weight Ratio (%)				
Silicon	100	75	70	65	60
Flame retardant	-	25	30	35	40

By using this specimen, the features on the heat and the flame of the fireproof board were analyzed. For the analysis, three tests on TGA, LOI, and Flame were performed. For TGA, The heat features of the material can be verified by reviewing the beginning temperature on the decomposition (standard of 5% loss) and the weight changes in analyzing the heat weight. LOI is a test for checking the concentration of oxygen to keep the combustion of materials. The higher concentration is, the better the performance of flame retardant exhibits. Lastly, flame test is one that checks the changes of the temperature on the rear of the specimen when it is burned while keeping the temperature of 1200 °C for 60 minutes by a gas torch. Hereby, it can be predicted how much heat can be prevented when the fireproof board is attached to concrete structures.

III. Results and Discussion

A. Silicon compounds and flame retardants

Silicon compounds have the Si-O structure where the silicon and the oxygen atoms cross each other. As various silicon compounds can be produced by adding or polymerizing several kinds of organic bodies, it is possible to have wide applications in different forms and multipurpose. Silicon compounds generally have outstanding features such as heatresistant, flame resisting, eco-friendly, nontoxic, and oxidation stability. Especially in the case of the silicon rubber to us in the research, it can be manufactured in accordance with the practical uses because it contains silica. In view of these features, it is clear to recognize that silicon compounds are highly practical in applying them at high temperature.

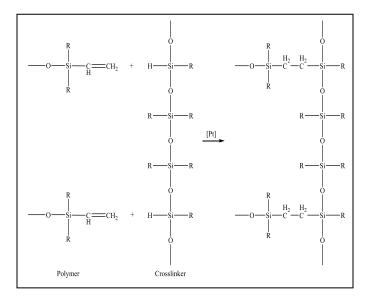


Fig. 1 Schematic illustration of chemical reaction of poly (dimethylsiloxane) rubber

Those flame retardants such as ATH, MDH, and MC added to this silicon base have features in common which they are non-toxic and stable in heat and exhibit the performance of flame retardants through the mechanism like the figure below.

$$2AI(OH)_3 \xrightarrow{180 \sim 200^{\circ}C} AI_2O_3 + 3H_2O - 298kJ/mol$$

$$Mg(OH)_2 \xrightarrow{332^{\circ}C} MgO + H_2O - 328kJ/mol$$

Fig. 2 Decomposition mechanism by heat for ATH and MDH

B. Feature Analysis

1) Feature analysis on heat (TGA)

After taking 20~30mg of each specimen to heat it from 25 up to 900°C at the speed of 20°C/min under rapid thermal anneal, the analysis results of the diminished weight have



come out as follows as the temperature changes proceed.

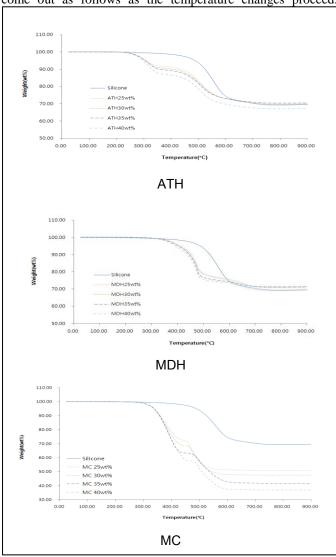


Fig. 3 Result graphs on the feature analysis on heat

As the heat analysis of the specimen with flame retardant starts at lower temperature than pure silicon compounds, the thermal stability is smaller than the pure silicon compounds. However, this is caused by the endothermic reaction of each flame retardant which is kind of decomposition reaction because flame retardants starts heat analysis first, faster than silicon after absorbing necessary heat. Superficially, it can be judged that heat stability is lower but it showed that the performance of flame retardant gets improved. Each specimen has showed over 70% of last residue except for MC, the organic compound. Therefore, it is judged that there is no problem about the performance of fire resistance due to diminished weight of the board in the event of a fire. In case of flame retardants, ATH and MDH except for MC, the specimen mixed with weight ratio 35% exhibited the best character. Through this, it is verified that the combination and the manufacture of effective performance for fire resistance depend on the mixture with specific weight ratio.

2) Analysis on flame retardants (LOI)

If the oxygen index obtained from LOI test is over the value 30, it is judged that it holds flame resistance. The result for the experiment is as follows.

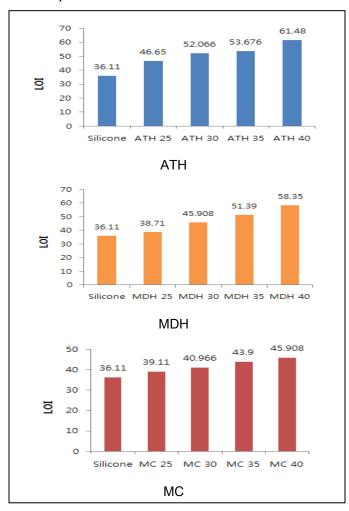


Fig. 4 Graphs on Limited Oxygen Index.

In case of pure silicon, the performance of flame resistance was verified at 36 and every specimen has obtained over 30 oxygen index in case that ATH, MDH, and MC are added. These values were all higher than pure silicon and verified that they obtained higher oxygen index as the content of flame retardants increases after each of them was checked. Especially, in case of ATH, when the same value of weight ratio was compared with that of other flame retardant, it was the highest enough to reach about 61 oxygen value which is the highest value of all the researches to confirm fire resistance and flame retardant when 40% of weight ratio was mixed.

3) Flame resistance test

In accordance with the standard (Management standards on high strength concrete columns and beams, Ministry of Land, Transport and Maritime Affairs, 2008), the temperature of main reinforced bars should be normally 538 $^{\circ}$ C, maximally 649 $^{\circ}$ C. Considering the spalling phenomenon at 250 $^{\circ}$ C ~350 $^{\circ}$ C, the test results for the performance are as follows.

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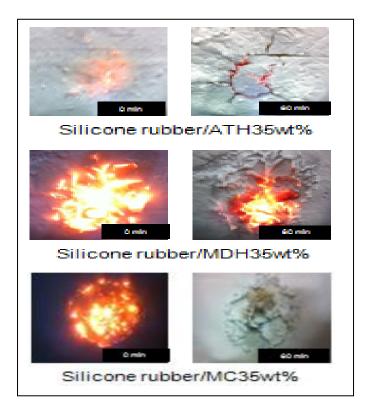


Fig. 5 Tests of fire resistance for each flame retardant (Left; Start, Right: End)

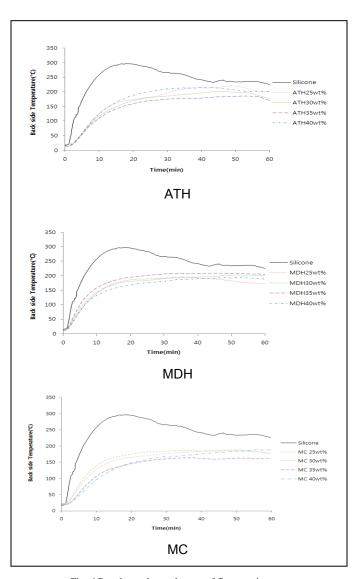


Fig. 6 Result graphs on the test of flame resistance

The pure silicon compounds indicate that the temperature of the rear specimen is about 300 °C. Though this doesn' t exceed the average and the highest temperature of the main reinforced bars, it is certainly hard to consider them fire resistant since it is within the range of temperature $250\,^{\circ}\text{C}\sim\!350\,^{\circ}\text{C}$. However, in case that the flame retardants was added, it is judged that the temperature of main reinforced bars and explosion prevention can be satisfied, considering the temperature of the rear specimen reached $200\,^{\circ}\text{C}$ on and off. As for the specimen with ATH 250wt%, it recorded $220\,^{\circ}\text{C}$ on the reverse side which is the highest among the specimens but it was confirmed that every specimen hold very high heat blocking ability and control of temperature transmission considering 1,200 °C applied to them.



iv. Conclusion

In order to figure out the possibility as a fire-resistant board of silicon compounds added with fire retardants, TGI, LOI, and Flame tests were performed and analyzed. Hereby, application probabilities, improvement of fire retardant performance and silicon compounds as a material for a fire-resistant board were verified. To put all the small bottom lines together is as follows.

- 1) In the flame test, the rear temperature was around 164.3~220°C, which is the temperature range the influence of concrete explosion and main reinforced bar can't reach. So it is judged that the performance of fire resistance can be secured by controlling heat transmission through the fireproof board.
- 2) Nitrogenous flame retardant, MC is an organic flame retardant different from nonorganic ones; ATH and MDH. It was all combusted by heat in the TGA test and showed very low final residue. Organic flame retardant was verified not to be appropriate to apply it to silicon based fireproof boards.
- 3) ATH in the TGA test had the lowest temperature in beginning temperature of heat analysis but it is judged to be effective to be applied to fireproof boards with 70% of final residue, while in the LOI test, it showed the highest oxygen index to confirm its high resistance against combustion.
- 4) In those two tests except for TGA test, all the specimens added with flame retardants had as high oxygen index as over 30 and rear specimen temperature of 250°C. Seen these facts, it is confirmed that silicon compound with flame retardants is more effective than pure one to improve the performance of fire resistance and so concrete structures are estimated to prove even more excellence to fire resistance when they are applied.

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