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An Experimental Study on Sodium-Concrete Reactions

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Abstract

A sodium-concrete reaction facility with a test chamber of $0.226m^3$ (ϕ 0.6m × H0.8m) was constructed to carry out experiments of sodium-concrete reaction which might take place in sodium metal fast-breeder reactor. Utilizing this facility, several experiments were conducted to closely examine the characteristics of sodium-concrete reactions under different conditions : Sodium mass = 100, 250g ; Sodium temperature = 450, 550, 650℃ ; Concrete age = 30, 45, 50, 90days. Our experiments show that the amount of the H_2 generated by sodium-concrete reaction has increased up to its flammable range as the amount of spilled sodium and its temperature have increased. The maximum hydrogen concentration was 31mol% at the concrete age of 30days, sodium temperature = 550℃, and sodium mass = 250g. The major components of sodium-concrete reaction products were also determined as Na_2SiO_3 and $NaAlO_2$.

1. Introduction

Concrete is a common construction material in liquid metal fast breeder reactor (LMFBR) facilities. Although the probability of a large spill of sodium directly on to a concrete surface of LMFBR facilities is very low, an evaluation of possible interactions resulting from such a hypothetical event is very important in their safety analysis because the LMFBR licensing procedures require that severe incidents, beyond the design basis, be analyzed.

If a sodium leakage leads to a hot sodium pool above a concrete surface, the following behavior must be taken into account:

- the reaction between the free water in the concrete and the sodium.
- the reaction between the chemically bound water and the sodium.
- the reaction between the concrete aggregates and the sodium.

The above behavior can be expressed by the

Key words : Sodium safety, Sodium technology, Sodium-concrete reaction, Sodium-concrete tests

Table 1. Classification of Concrete Aggregates and Its Application[2]

Type of aggregates	Type of aggregates Major Components	Major Reaction Products with Sodium	Utilization of Reactor or Testing Laboratory
Basalt	SiO_2, Al_2O_3	Na_2SiO_3, $NaAlO_2$	FFTF[1)], U. S. A
Magnetite	Fe_3O_4	Na_2O, FeO, Fe	HEDL[2)], U. S. A
Limestone	$CaCO_3$, $CaMg(CO_3)_2$	CaO, MgO, Na_2CO_3, C	CRBR[3)], U. S. A CEA, France
Granite	SiO_2, CaO	Na_2SiO_3, Na_2CaSiO_4	PFR[4)], U. K
Serpentine	$Mg_3Si_2O_5(OH)_4$	MgO, Na_2SiO_3	KfK, Germany
Graywacke	SiO_2	Na_2SiO_3	Oarai, Japan

1) Fast Flux Test Facility 2) Hanford Engineering Development Laboratory

2) Clinch River Breeder Reactor 4) Protype Fast Reactor

following reactions :

$$Na + H_2O \rightarrow NaOH + \frac{1}{2}H_2, \ \Delta H_{1100K} = -38Kcal \quad (1)$$

$$2NaOH + SiO_2 \rightarrow Na_2SiO_3 + H_2O, \ \Delta H_{1100K} = -122Kcal \quad (2)$$

$$2NaOH + Al_2O_3 \rightarrow 2NaAlO_2 + H_2O, \ \Delta H_{1100K} = -101Kcal \quad (3)$$

$$4Na + 3SiO_2 \rightarrow 2Na_2SiO_3 + Si, \ \Delta H_{1100K} = -24Kcal \quad (4)$$

Consequently, these sodium-concrete reactions are sources for both energy and hydrogen releases to the containment. Both the energy released and hydrogen generated may contribute to the challenge of LMFBR containment integrity.

Numerous studies have been conducted to characterize sodium-concrete reactions in the United States, France, Germany, United Kingdom and Japan with LMFBR development [1, 3-9]. They have covered basic aspects and characteristics of sodium-concrete reactions and provided experimental data for an evaluation of the potential consequences of sodium-concrete reactions. They were reviewed recently in detail [2]. In their study the concrete aggregates in the reactor construction building are often found to be different from each country and that the results of sodium concrete reactions were different.

Table 1 summarizes major components of concrete aggregates and their reactor products with sodium.

The reactor safety concern related to sodium technology is an essential part of LMFBR development. Therefore, further sodium-concrete reaction studies are in progress in France and Japan to improve their reactor safety. As part of accumulation of sodium safety technology for LMFBR here in Korea, sodium-concrete reaction studies were carried out in our laboratory. A small sodium-concrete reaction test facility was set up and operated under different operating conditions. And the characteristics of sodium-concrete reactions based on the experimental results are analyzed for establishment of sodium safety technology in this field.

2. Experiment

2.1. Sodium-Concrete Reaction Facility

The experimental test facility consists of a test chamber, a sodium heating tank, a scrubber, a control panel, and a data acquisition system as shown in Figure 1. Sodium-concrete reaction tests

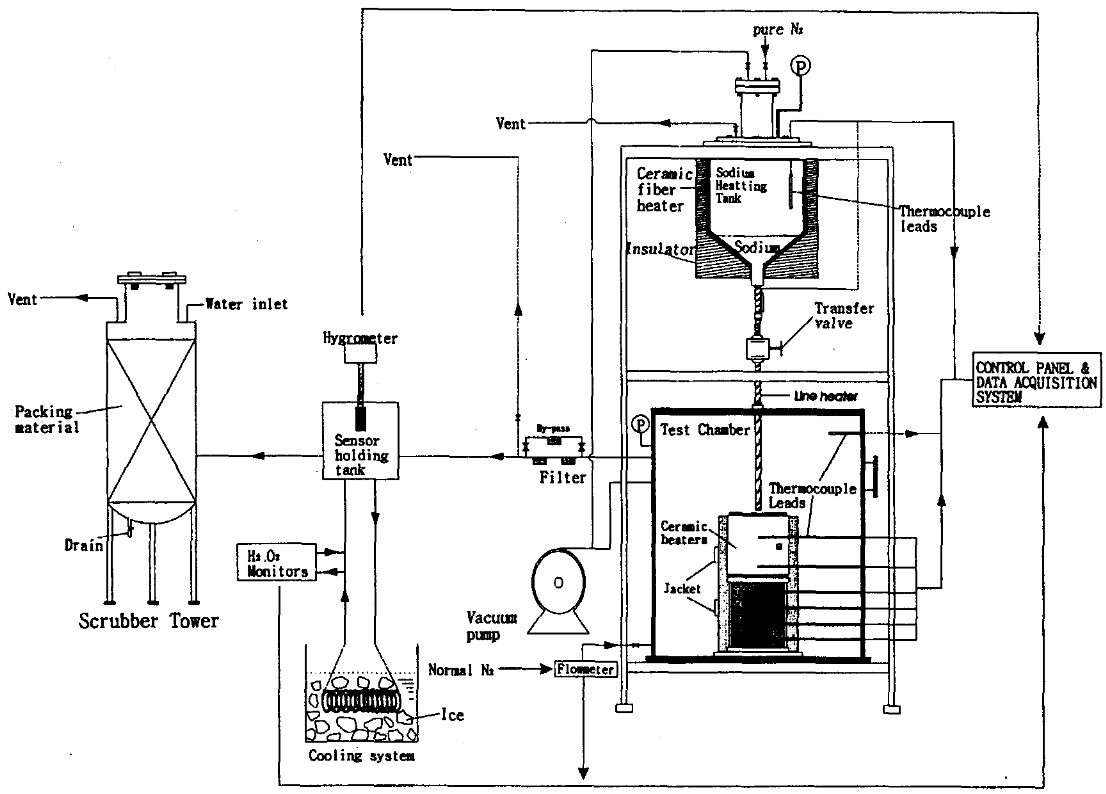
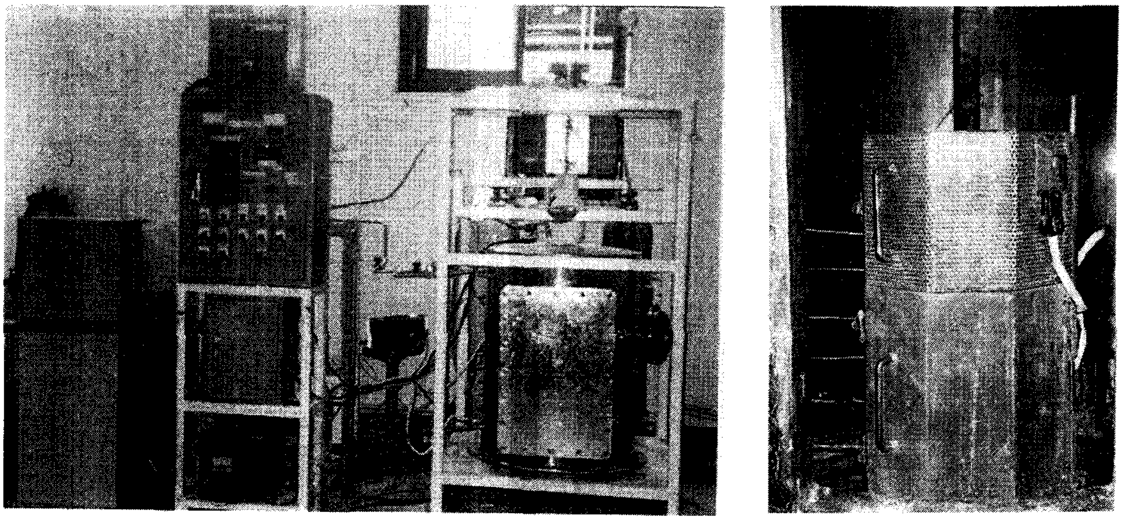


Fig. 1. Sodium-Concrete Reaction Test Facility

were performed in a 0.226㎥ (0.6m×H 0.8m) stainless steel test chamber in which the concrete test article is introduced. The sodium heating tank with 0.0033m^3 capacity was designed to heat sodium and supply to the sodium-concrete reaction test chamber at a certain temperature. The gas scrubber is a packed tower which is filled with water. Sodium-concrete reactions produce aerosol particles and hydrogen from interaction between Na and H_2O in concrete. These are scrubbed, filtered and discharged to a stack in the gas scrubber. The control panel is designed to control the temperature of sodium heating tank and to indicate pressure, oxygen concentration, temperature in the chamber, and the temperature distribution in the concrete test article.

Data acquisition system measures pressure, temperatures, hydrogen, oxygen, humidity, and gas flow rate. The signals from pressure transducer, thermocouples, H_2/O_2 meter, hygrometer and gas flow meter are converted into digital data a 12bit A/D converter(Real Time Devices, AD2110) with through put of 40㎑ which is controlled by the personal computer. The total sampling number was eleven : seven from temperature sensor, one from oxygen monitor, one from hydrogen monitor, one from pressure transducer, and one from hygrometer. As all measurement variables were changed abruptly as soon as melted sodium was injected into the concrete sample, their sampling rate was initially set up as 10HZ. And the rate was reduced to 1HZ



(a) Overall View (b) Inside of Test Chamber

Fig. 2. Pictures of Sodium-Concrete Reaction Facility

Table 2. Measurement Sensors of Data Acquisition System

Classification	Maker & Model	Output Signal, Type	Error range
Oxygen monitor	Hi-Teck, KG850, 810-0019	0 - 1 V	± 0.1%
Hydrogen monitor	Hi-Teck, KG850, 810-0019	0 - 1 V	± 0.2%
Hygrometer	ShinHo, IO696139	4 - 20 mA	± 0.5%
Pressure Transducer	Trans Metrics, P21AA, 83906	0 - 5 V	± 0.1%
Thermocouples	Kusam, K-Type(1/8")	4 - 20 mA	± 0.5%

after 30minute and to 0.1Hz after 1hr. The digital data are stored in the PC and analyzed afterward. The overall view and the interior of the test chamber of the sodium-concrete test facility are shown in Figure 2. And the specifications of the measurement sensors were listed in Table 2.

2.2. Concrete Test Articles

A concrete test article assembly was constructed in two separate sections : concrete article and sodium reservoir. The concrete article were prepared by pouring 0.00196m^3 of concrete into a mild steel cask. The sodium reservoir section was formed separately and joined to the concrete sections with bolts at the mating flanges. Four thermocouples in the concrete section and two thermocouples in the sodium reservoir section were installed at locations shown in Figure 3. Tubular electric heaters were attached to the exterior of the sodium reservoir immediately above the concrete level. Insulation materials surrounded the concrete and sodium sections of each vessel.

The constituents of concrete which was used in this study are summarized in Table 3. These constituents are similar to the PFR concrete in the

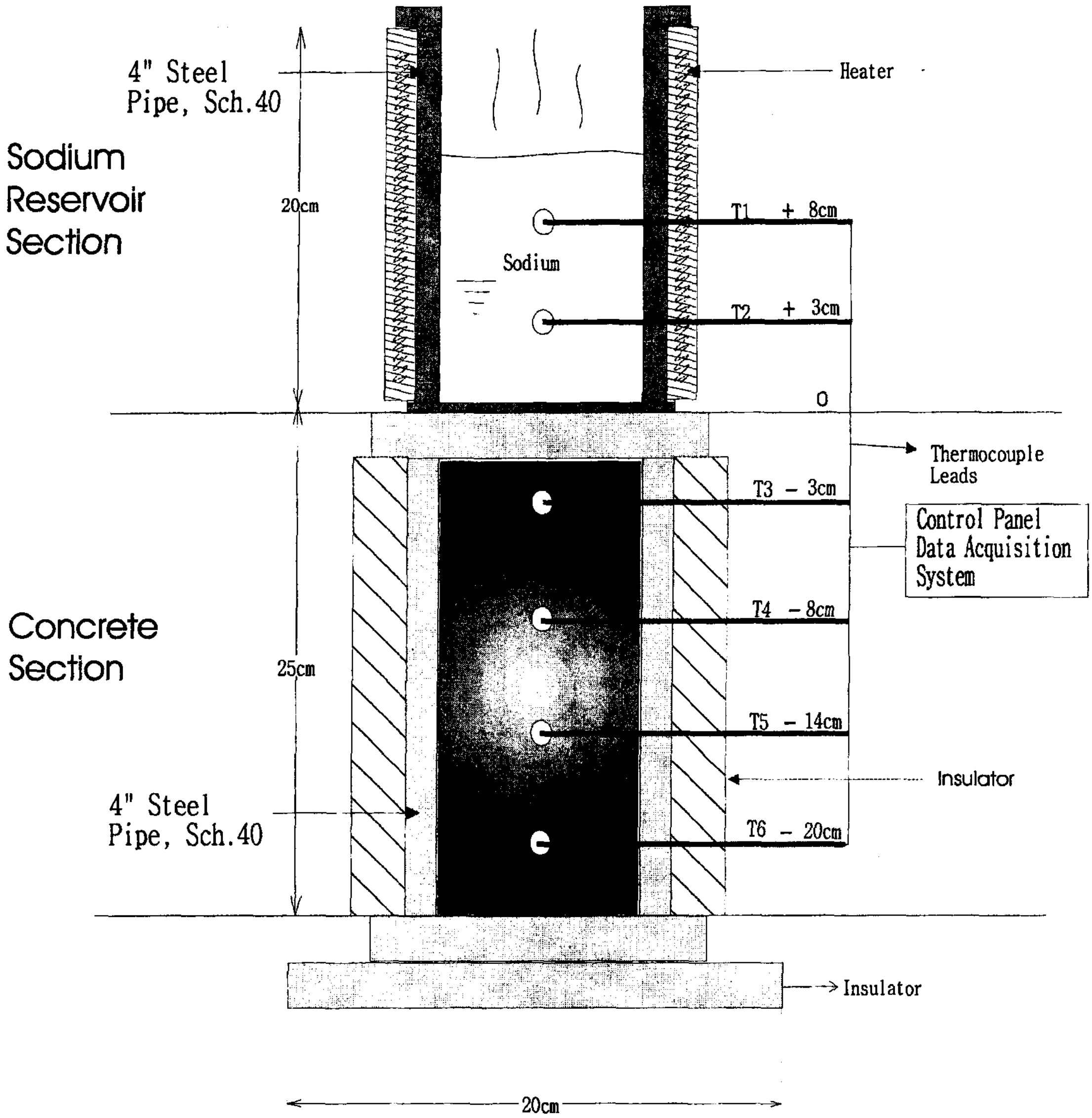


Fig. 3. Concrete Test Article Assembly

Table 3. Composition of Unmixed Constituents for the Concrete Sample

Constituent	Portland cement[1)]	Aggregate[2)]	Sand[3)]	Water
weight (kg)	6.24	18.80	12.48	2.48
weight percentage (%)	15.6	47	31.2	6.2

1) Ordinary type (KS L 5201)   2) Produced by Kyongki province

3) Jumunjin sand grade

United Kingdom [10].

The concrete mixture was prepared by mixing Portland cement, aggregate, sand, and water by Korean standard specification for concrete manufacturing. After the concrete mixture was poured in the concrete test article, the article was shaken on a shaker table for a few minutes and the top surface smoothed with a wood trowel. The concrete sample was hardened and cured for more than 30 days before the experiment. The concrete strength approaches 90% of its maximum value at one month's curing time. Thus, the ages of the concrete sample were selected in the range of 30 to 90days to observe the effect of concrete ages on sodium-concrete reaction in our experiment.

2.3. Test Procedure

The sodium-concrete reaction tests were performed in the test facility shown in Figure 1. Before starting the experiments, it is necessary to confirm the proper operation of control panel, data acquisition system, and measurement instruments. After confirmation of proper operation of all systems, instruments of the facility and various experimental conditions of sodium-concrete reaction are adjusted. The basic test procedure is as follows :

- Install the concrete test article assembly in the test chamber.
- Put the weighed solid sodium in the sodium heating tank.
- Purge the sodium-concrete test system with nitrogen.
- Melt and preheat sodium in the sodium heating tank at certain experimental temperature.
- Start the data acquisition system for measurement of hydrogen and oxygen concentration, pressure, humidity, and temperatures in the test chamber and the concrete test article
- Set the rate of nitrogen purge in the test chamber.
- Dump the heated sodium into the vessel from the sodium heating tank.
- Heat the sodium reservoir vessel at experimental temperature.
- Stabilize the system at desired level for required time.
- Stop electrical heating of sodium reservoir vessel, let system cool down for certain period of time, and stop data acquisition system.
- Disassemble the concrete test article assembly.
- Conduct a visual examination and take a photograph, and perform chemical analysis of sodium-concrete reaction products.

Experimental conditions and some results of sodium-concrete reaction tests are shown in Table 4. The hydrogen concentration was obtained through on-line hydrogen monitor and the amount of total H_2 moles generated was calculated from the on-line hydrogen concentration and gas flow rate. In order to analyze samples of sodium-concrete reaction products, XRD (X-ray Diffractograms, Rikaku/MAX-3C) filtered CuKα_1 radiation (λ = 1.541 Å) being used at a scanning rate of 2°/min and SEM-EDS(Scanning Electron Microscope - Energy Dispersive System, HITACHI 2700) were utilized.

And the reproducibility of sodium-concrete reaction experiments was quite good in our experimental range

3. Results and Discussion.

3.1. Influence of Experimental Variables on Sodium-Concrete Reaction Products

Different experimental variables in sodium-concrete reactions for this experiment are sodium reservoir heating temperatures, aging days of concrete test articles, and the amount of sodium injected in the test chamber. All experiments are

Table 4. Test Chamber(φ 0.6m × H0.8m) Experimental Conditions and Some Results of Sodium-Concrete Reactions.

Test No.	Concrete Age (Days)	Sodium Injection		Sodium Reservoir		N_2 Purge Rate (l /min)	Total H_2 Generated (mole)	Maximum H_2 Conc. (mol %)
		Mass(g)	Temp.(℃)	Heating Temp.(℃)	Heating Method			
1	30	250	500	No Heating		5	0.20mole	0.95
2	45	250	500	No Heating		5	0.18mole	0.89
3	30	250	500	550	①	5	4.15mole	31
4	45	250	500	550	①	5	Failure	Failure
5	45	250	500	550	②	5	2.20mole	12.5
6	45	250	500	550	①	5	2.16mole	15
7	45	100	500	550	①	5	1.77mole	5.2
8	45	250	500	450	①	5	1.98mole	3.5
9	45	250	500	650	①	5	3.95mole	24.8
10	50	250	500	550	①	5	2.35mole	13
11	90	250	500	550	①	5	2.22mole	14.4

① After sodium injection ② Before sodium injection

performed in an inert atmosphere filled with nitrogens. In the initial experimental work of sodium -concrete reactions, when there was no heating in the sodium reservoir after injection of sodium from the sodium heating tank, the amount of H_2 generated was found to be very small as shown in Table 4 (Tests 1 and 2). This explains that the heated sodium at a certain temperature cools down immediately after its injection in the test chamber. The sodium-concrete reaction is limited, and the maximum instantaneous H_2 concentration (0.89 ~ 0.95 mol%) was below the lower flammable limits of hydrogen(4.0 mol%).

Heating the sodium reservoir up to 100℃ for a few minutes before sodium injection was tried in Test 5 in oder to retard sodium cooling down suddenly. This method slowed down sodium cooling, but there was no difference in the amount of hydrogen generated in sodium heating methods between heating before and after the injection in the test chamber. This was implied that the free water on the surface layer of the concrete was almost dried up after its ageing period. Thus, the sodium reservoir was heated after sodium injection in the remaining experiments except test 5.

The experimental data for the operating condition of different sodium reservoir heating temperature and the same concrete age and sodium mass are shown in Figures 4 and 5. Figure 4 indicates that the temperature of the concrete test article attains a steady value within 200 minutes and the temperature of its bottom part approaches 100℃ regardless of sodium reservoir heating temperature. But, the interior temperature of the concrete test articles changed and the amount of H_2 generated varied depending upon sodium heating temperatures : 1.98, 2.16, and 3.95 moles of hydrogen were generated at sodium reservoir heating temperature of 450℃, 550℃, and 650℃, respectively. The maximum H2 concentration was 24.8 mol% at temperature 650 ℃ with concrete age of 45 days and sodium mass = 250g. And the rate of hydrogen generation due to sodium-concrete reaction was shown to be irregular and have two as shown peaks in Figure 5. These phenomena might take place because

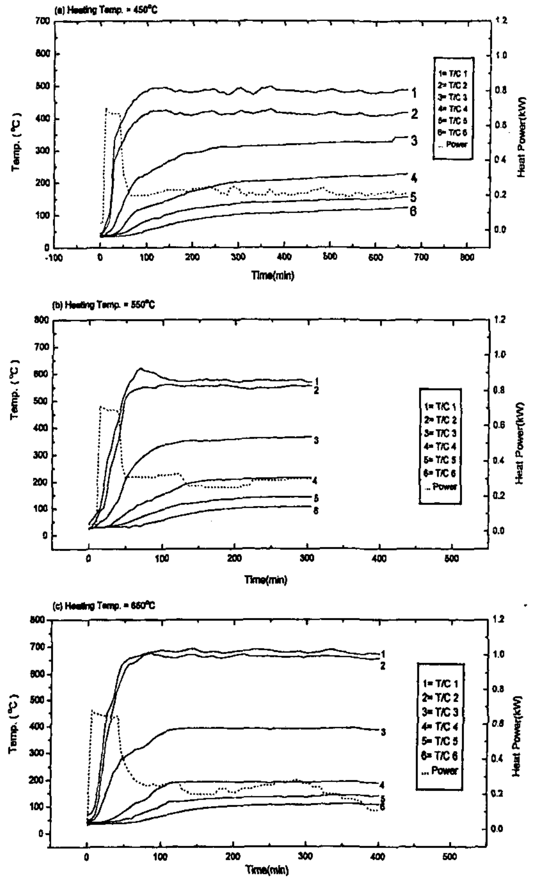


Fig. 4. Temperature Change of Test Article Assembly for Different Sodium Reservoir Heating Temp.-Exp. Conditions: Concrete Age = 45 Day, Sodium Mass = 250g

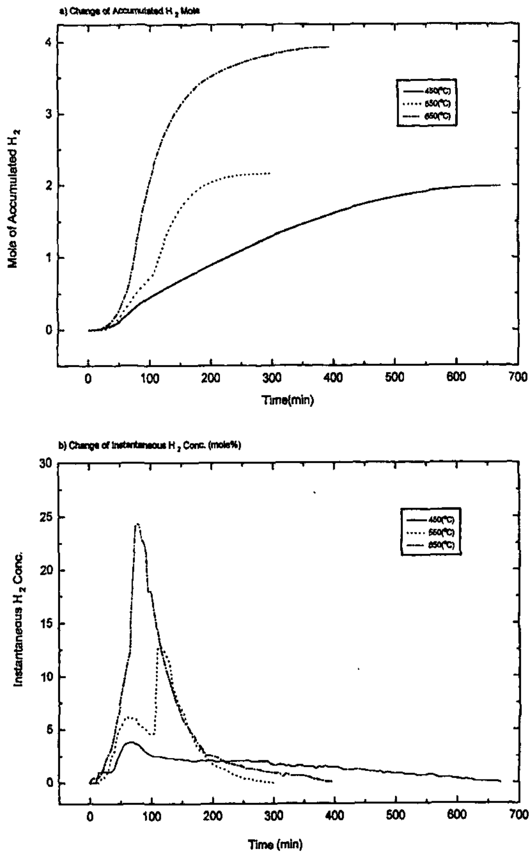


Fig. 5. Change of the Amount of H_2 Generated for Different Sodium Reservoir Heating Temp. - Exp. Conditions: Concrete Age = 45 Day, Sodium Mass = 250g

the concrete consists of heterogeneous materials and the temperature control of the sodium reservoir was not smooth. These kinds of as peak forms of H_2 generation rate were reported by McCormick et al [18] and Cherdron[19].

It is noticeable that the temperature difference between the locations 5 and 6 in comparison with other locations for all heating temperatures(450, 550, 650℃) was quite small. Such a result might be generated by heat loss at the bottom side of the concrete sample even though insulation materials of ceramic fiber was covered with the sample' s bottom.

Experimental data for the operating conditions of different concrete age at constant sodium heating temperature and sodium mass are shown in Figures 6 and 7. The temperature change of concrete test article had a similar curve pattern, but the amount of H_2 generated changed by the concrete age. The amount of H_2 generated was maximum at the concrete age of 30days and it became almost constant after concrete age of

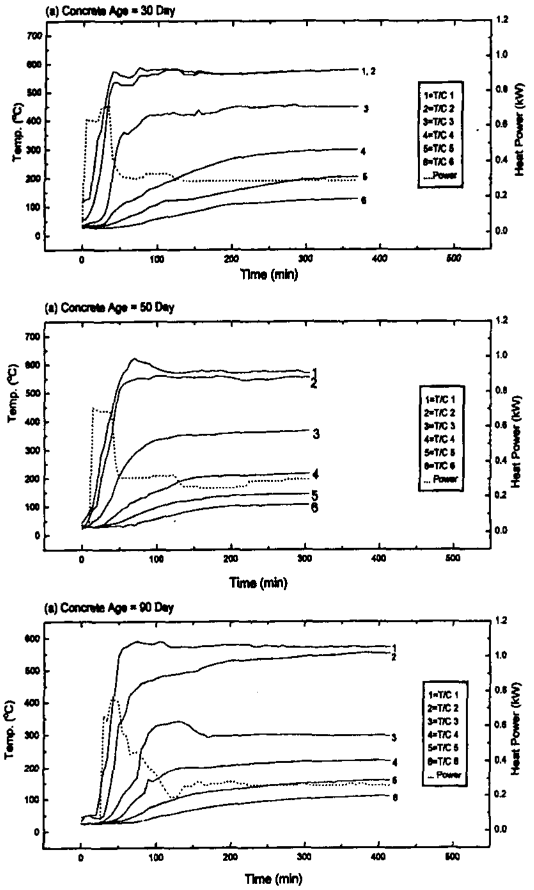


Fig. 6. Temperature Change of Test Article Assembly for Different Concrete Aging Temp. - Exp. Conditions: Sodium Mass = 250g, Heating Temp. = 550℃

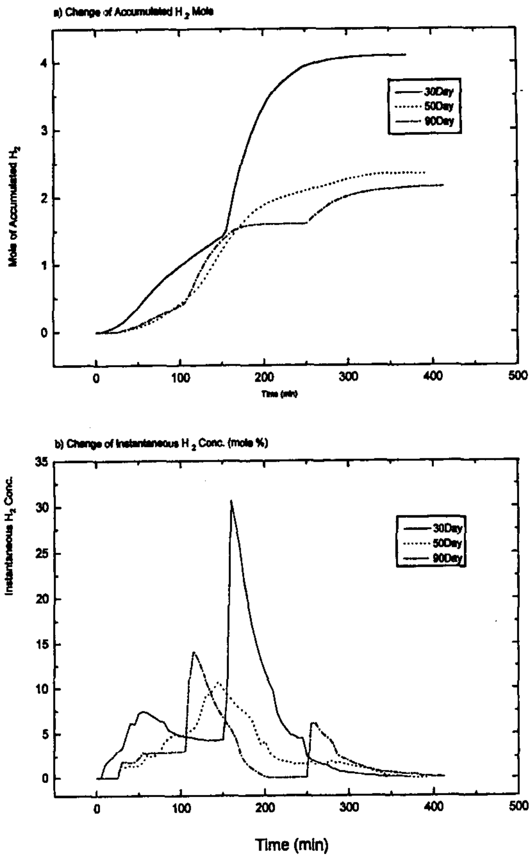


Fig. 7. Change of the Amount of H_2 Generated for Different Concrete Aging Time - Exp. Conditions: Sodium Mass = 250g, Heating Temp. = 550℃

45days. It is observed that the amount of H_2 generated by sodium-concrete reaction was approximately 2.16~2.35 moles for the concrete ages longer than 45 days at sodium mass = 250g, and sodium heating temperature = 550℃ as shown in Table 4.

Experimental data of sodium-concrete reaction for different sodium mass are shown in Figure 8. It indicates that as the initial amount of sodium mass increases, total moles of H_2 generated and the hydrogen concentration slightly increases.

The lower and upper flammable limits of hydrogen concentration are 4.0 and 75 mol% in air, respectively[11]. In most of our experiments, the hydrogen concentrations generated were within the flammable limits which their maximum concentration reached up to 31 mol%. It is expected that the hydrogen explosion reaction will take place if our experiments would be done in open air, not in the nitrogen atmosphere. Thus, this proves that the hydrogen explosion accident may occur if the sodium-concrete experiments were carried out in the open air.

In consideration of the safety measures against a

Table 5. Compositions of Different Types of Concrete

Types of Concrete	Composition						
	SiO_2	CaO	Al_2O_3	FeO	MgO	K_2O	others
Magnetite	2.5	6.9	0.4	89.8	0.1	0	0.3
Limestone	11.9	70.6	3.9	3.3	7.7	1.4	1.2
Basalt	58.2	16.2	11.2	7.0	3.4	3.6	0.4
This study	52.3	24.8	10.2	3.34	4.92	3.37	1.07

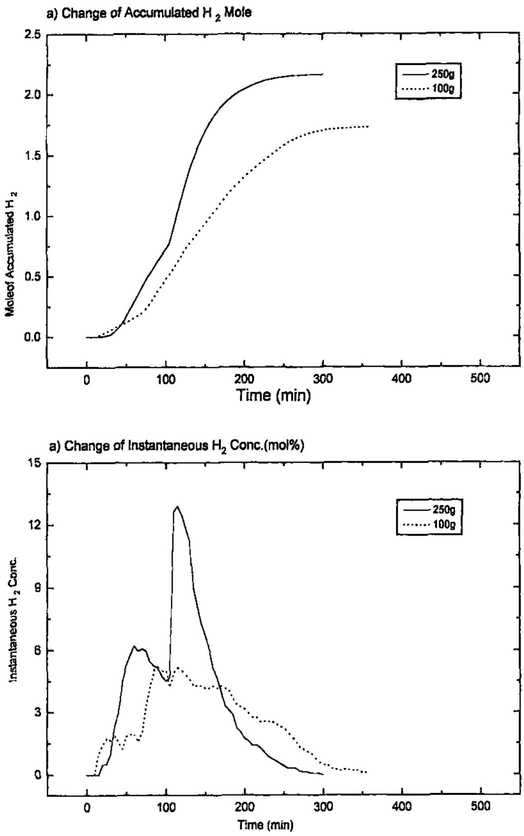


Fig. 8. Change of the Amount of H_2 Generated for Different Sodium Mass - Exp. Conditions: Cocrete Age = 45Day, Heating Temp. = 550℃

possible sodium-concrete reaction and H_2 generation due to a sodium leakage from the LMFBR facility, the steel liner has been covered on the concrete surface. However, it was recently proven that this safety feature was not enough to prevent sodium-concrete reaction in the Monju accident[9]. Thus, a inert atmosphere must be maintained for possible sodium leakage areas to prevent severe accidents in the LMFBR. The utilization of special chemicals- and heat-resistant concrete which do not react with sodium is also one of the methods to mitigate consequences of the possible sodium spill in the LMFBR facility [12-14].

3.2. Analysis of Sodium-Concrete Reaction Products

The compositions of concretes were analyzed by SEM-EDS as shown in Table 5. They are compared with other types of concretes. The compositions of the concrete used in the experiment are similar to basalt concrete of which SiO_2, CaO and Al_2O_3 are the major components [15].

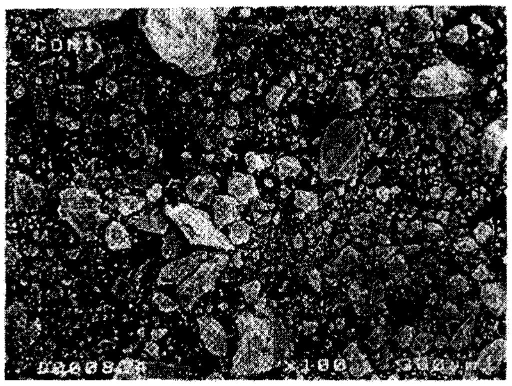
The components of sodium-concrete reaction products were detected by SEM-EDS as shown in Table 6. They are compared with fresh concrete used for this experiment. SEM-EDS measures approximate compositions of different metal oxide materials. The major composition of sodium-concrete reaction products is Na_2O as shown in Table 6. Na_2O may be found as the compound form of $Na_2SiO_3(Na_2O+SiO_2)$ or $NaAlO_2$ which are not distinguishable by SEM-EDS.

SEM pictures of concrete samples are shown in

Table 6. Compositions of Fresh Concrete and Sodium-Concrete Reaction Products

State	SiO_2	CaO	Al_2O_3	FeO	MgO	K_2O	Na_2O	others
fresh concrete	52.3	24.8	10.2	3.3	4.9	3.4	0	1.1
reaction products	23.1	7.6	5.3	1.2	4.5	3.2	54.5	0.6

a) Before Sodium-Concrete Reaction



b) SEM Pictures of Concrete Samples

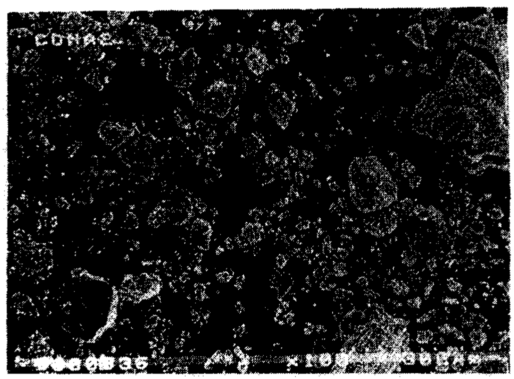


Fig. 9. SEM Pictures of Concrete Samples

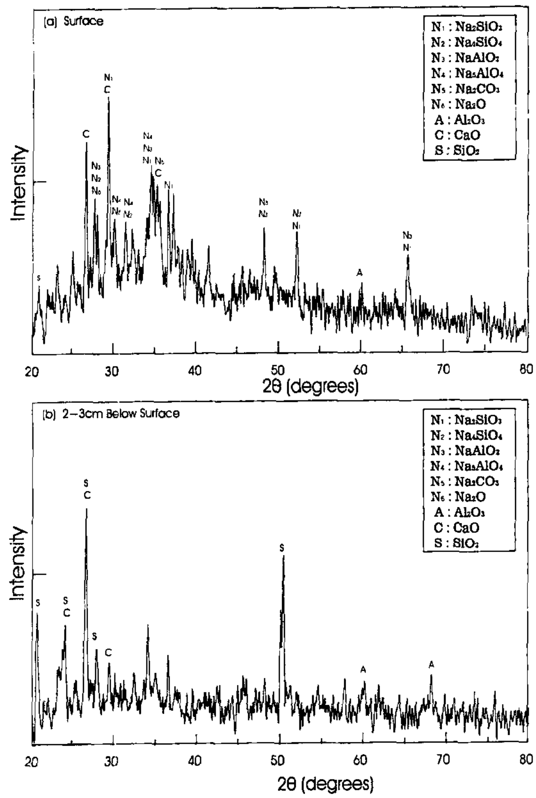


Fig. 10. X-ray Diffractograms of Sodium-Concrete Reaction Products - Exp. Conditions: Concrete Age = 30 Day, Sodium Mass = 250g, Sodium Reservoir Heating Temp. = 550℃

Figure 9 before and after the sodium-concrete reaction. The black portion in Figure 9-b) is the sodium oxide compound. The XRD graphs of concrete and reaction products are shown in Figure 10. Major compositions of experimental concrete used are SiO_2, CaO, and SiO_2 like conventional concrete. However, the sodium-concrete reaction products are found to be Na_2SiO_3 and $NaAlO_2$ which may be produced from reactions (1) to (4)[16, 17]. Hassber et al. reported that most of reaction products were Na_2O and Na_2SiO_3[15]. However, Barker et al. showed that the reaction products were Na_2SiO_3 and Na_2CaSiO_4[16]. Thus, it can be concluded that the reaction products were mainly determinated by the concrete aggregates.

4. Conclusions

Sodium-concrete reaction experimental studies have been performed in a small test facility. The hydrogen concentrations generated by sodium-concrete reaction tests in most of our experiments ranged within the flammable limit of hydrogen. Their maximum concentration was 31 mol% at concrete age of 30 days, sodium temperature = 550℃, and sodium mass = 250g. Total moles of hydrogen generated by reactions increased up to its flammable range as the amount of spilled sodium and sodium heating temperature increased. The concrete used for this experiment is the conventional concrete in Korea with similar composition to basalt concrete. And the major components of sodium-concrete reaction products in our experiment were found to be Na_2SiO_3 and $NaAlO_2$ as compared to other experimental data of Na_2O and Na_2SiO_3 or Na_2SiO_3, Na_2SiO_4 and Na_2CaSiO_4

Further study on sodium-concrete reaction modeling and development of chemicals- and heat-resistant concrete are necessary for the establishment and improvement of safety measures against unwanted sodium-concrete reactions in hypothetical accidents of the LMFBR.

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