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Large-Scale Exploratory Tests of Sodium/Magnetite-Concrete Interactions

MASTER

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Sandia National Laboratories
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April, 1983

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Abstract

Two large-scale tests examining the interaction between molten sodium at 825 K and magnetite concrete were performed. In the first test, bare magnetite concrete was exposed to the sodium and in the second test the concrete was partially protected by a flawed steel liner and a layer of dense silica firebrick. The results demonstrated that energetic chemical reactions can occur between sodium and magnetite concrete. In the first test, reaction zone temperatures exceeded 1150 K and the maximum heat-generation rate was $\sim 1.3 \times 10^5 \text{ J/m}^2\text{-s}$. The maximum observed penetration rate was 2.5 mm/min. In the second test, the silica firebrick provided little protection for the underlying concrete. The reaction debris that formed between the steel liner and the concrete caused gross deformation of the liner as well as extensive cracking of the reinforced concrete.

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Introduction

Liquid metal fast breeder reactors (LMFBR) use molten sodium as a coolant in the primary cooling loop. For certain postulated accident situations, molten sodium at temperatures in excess of 825 K could come into contact with the concrete used in construction of the containment building. The interaction between the molten sodium and concrete could threaten containment integrity because of the effects of the chemical attack of the sodium upon the concrete or by the effects that the chemical reaction products have on the containment structures. These include such phenomena as hydrogen generation, heat generation, and aerosol production.

Sandia National Laboratories, Albuquerque (SNLA), under its Advanced Reactor Safety Research Program, has conducted studies of the interaction between molten sodium and concrete for conditions similar to hypothetical reactor accident conditions. The objective of this program is to establish a data base for sodium/concrete interactions which can support the necessary safety studies, risk analyses and environmental impact investigations pertinent to LMFBR accident conditions. The study has included three types of concretes which differ principally in the aggregate used. The aggregates are limestone (calcite), basalt, and magnetite. Concretes using basalt and limestone aggregates are of interest to the program because they are used as structural concretes in the containment building. Companion reports have outlined the results of large-scale tests with basalt concrete¹ and limestone concrete².

The present report presents the results of two large scale tests conducted with magnetite concrete. Magnetite concrete is of interest because it is used where biological shielding is desired. For certain hypothetical accident situations, molten sodium could contact the magnetite concrete as well as the structural concrete. The magnetite concrete used in the present tests meets the specifications of the magnetite concrete used in the Fast Flux Test Facility (FFTF) in Hanford, Washington, where it serves a dual purpose as a structural member as well as a biological shield (Figure 1).

The two magnetite tests reported here were part of a series of large-scale tests.^{1,2} In a large-scale test, a preheated sodium charge of approximately 100 - 200 kg is transferred rapidly into the concrete test article which is at ambient temperature. The test article is generally in the shape of a large cylindrical crucible with a cylindrical cavity to contain the molten sodium. The amount of sodium,

Figure 1. FFTF Reactor Cavity Structures.

and the size and shape of the crucible and cavity depend on the particular test being performed. The crucibles used in the present magnetite concrete tests weighed approximately 6000 kg. During the test, electrical pool heaters were used to simulate the decay heat from core debris which, in an actual accident situation, would maintain an elevated pool temperature. The experiments were monitored for gas and aerosol evolution, temperatures in the sodium pool and concrete, and erosion rate of the concrete. After the test, the reaction debris was chemically analyzed to help determine which important chemical reactions occurred during the test.

The two magnetite concrete tests were tests numbers 14 and 15 of the overall large scale test series at SNLA. They were performed specifically to examine the magnetite concrete used in the FFTF; no further testing of magnetite concrete is planned. Test No. 14 incorporated a bare magnetite concrete crucible with a cylindrical cavity and a relatively shallow sodium pool (123 kg of sodium). The test was designed to examine the interaction between molten sodium at 825 K and unprotected magnetite concrete. Test 15 was more complex and was designed to test specific FFTF design features. The test incorporated a square crucible cavity with a flawed steel liner and used 243 kg of sodium. The purpose was to examine the attack of vertical magnetite concrete walls and insulating siliceous firebrick which were partially protected from the sodium by a steel plate containing flaws.

Test Configuration

Test Site Facility

A schematic of the large scale test facility is shown in Figure 2. (Detailed descriptions of this facility have been presented elsewhere.^{1,2,3}) The facility is capable of preheating as much as 250 kg of sodium to 1025 K and transferring this sodium rapidly into large concrete test articles in the form of crucibles. The sodium is obtained in commercially available 55 gallon drums. It is melted and heated to 400 K in the drums before being transferred to the main high temperature heating tank where it is brought to the desired pretest temperature. Rapid transfer of the molten sodium from the high temperature heating tank to the test article is accomplished with a pneumatically operated rupture diaphragm valve. The valve has a conical stem and seat assembly opposite the rupture diaphragm section and is capable of being positively seated after transfer of the sodium. 250 kg of sodium can be transferred to the test article in less than one minute. After transfer of the sodium is complete, the valve is reseated so that no gaseous

Figure 2. Large Scale Sodium/Concrete Test Facility.

Figure 3. Top Hat Configuration.

reaction products can escape into the high temperature heating tank. A steel shield commonly known as the "top hat" fits on the top of the test article and isolates the sodium/-concrete reaction from the atmosphere (Figure 3). The top hat has feedthrough fittings for thermocouples which are used to monitor temperatures in the sodium pool and vapor space as well as electrical feedthroughs for power to the immersion heaters. The top hat also contains the necessary purge and vent lines which are used to bring inert gas into the test article and to allow the gaseous reaction products to escape. Particle separators and gas scrubbers are used to clean the vent gas before it is returned to the atmosphere. Immersion heaters are used in the sodium pool to simulate the decay heat from the core debris. The heaters have a maximum power rating of 50 kW.

Crucible instrumentation includes thermocouples embedded at various locations in the concrete to measure temperature profiles during the test and ultrasonic transducers which are used to measure the penetration rate with pulse echo techniques.^{4,5} Typically, between 15 and 20 thermocouples are embedded in the concrete crucible. Top hat instrumentation utilizes four thermocouples for measuring temperatures in the sodium pool and vapor space. Pressure transducers (for measuring pressure transients during the test), gas flow meters, and gas composition analyzers are mounted directly on the top hat or in the vent line which goes from the top hat to the separators and gas scrubbers. Aerosol sampling devices are connected to the vent line of the top hat outside of the test chamber. Automatic data acquisition during the test is accomplished by using a computer controlled data acquisition system.²

Test Articles

The crucibles were placed (cast) using magnetite aggregate obtained from FFTF sources and mixed in accordance with the FFTF concrete mix formulation. The mix specifications for the concrete are listed in Table 1. Crucibles were placed at the Eric H. Wang Civil Engineering Research Facility at the University of New Mexico. Crucible dimensions are listed in Table 2. General crucible construction details are shown in Figure 4. The crucibles were placed inverted, e.g., the cavity was upside down during placing. Reinforcing bars were used in the concrete around the outer sides and bottom of the crucible. The bars were located approximately eight cm from the concrete surface as shown in Figure 4.

To assure proper moisture content and strengths typical of reactor substructures the crucibles were cured under ambient conditions for a minimum of 90 days prior to the

TABLE 1

Magnetite Concrete Mix Specifications

	<u>kg/m³</u>	<u>lb/ft³</u>	<u>%</u>
Portland Cement, Type II	376.4	23.5	10.1
Water	185.8	11.6	5.0
Pozzalan	25.6	1.6	0.7
Aggregate (Magnetite), 3/4 in.*	1723.3	107.6	46.2
Sand (Magnetite)*	1419.0	88.6	38.0
	-----	-----	-----
	3730.1	232.9	100.0

*Central Premix Cement Co., Spokane, WA.

TABLE 2
Test Configurations

<u>Item</u>	<u>Test Number</u>	
	<u>14</u>	<u>15</u>
Crucible:		
Pour Date	11/10/78	1/22/79
Test Date	3/28/79	9/19/79
Outer diameter, cm	198	160
Height, cm	66	99
Cavity diameter, cm	122	84 (square)
Cavity depth, cm	28	61
Test Atmosphere	Ar	Ar
Liner:	None	
Height, cm	--	61
Length, cm	--	82
Width, cm	--	67
Slot size, cm	--	0.6 x 31
Firebrick:	None	
Wall A	--	None
Wall B	--	2 courses, dense (15.3 cm total thickness)
Sodium:		
Mass, kg	123	243
Initial temperature, K	915	975
Initial pool depth, cm	13.5	55
Heater set point, K	825	825
Results:		
Energetic reaction	Yes	Only in firebrick
Sodium consumed, kg	A11	~175
Max. erosion, cm	11	<1
Cracking of crucible	Yes	Yes

**TRANSPARENT VIEW SHOWING
REBAR, CHANNELS , AND ANCHORS**

Figure 4. General Crucible Construction.

TABLE 3

Concrete Compressive Strengths

<u>Cure Time</u>	<u>Test 14</u>		<u>Test 15</u>	
	MPa	(psi)	MPa	(psi)
7 day	23.9	(3468)	--	(--)
14 day	27.2	(3944)	24.0	(3485)
28 day	40.4	(5854)	32.1	(4652)
90 day	48.0	(6962)	35.2	(5111)
Test day	48.8	(7075)	48.5	(7039)

test. Strengths for the magnetite concretes used are listed in Table 3.

The cavity configuration for Test 14 was a right circular cylinder. Thermocouples were embedded in the crucible at the time of placing. The positions for these thermocouples are shown in Figure 5.

Test 15 was designed to examine specific engineering features used in FFTF. For that reason the cavity configuration in Test 15 was square, (e.g., straight vertical walls) rather than cylindrical. The cavity geometry used in Test 15 is shown in Figure 6. The test was designed to use a flawed steel liner with the flaws (0.6 cm x 30.5 cm slots) cut in opposing vertical sidewalls. This permitted sodium to contact only the two opposing walls. The remaining two opposing walls and the floor of the cavity were not used in the test. The floor was protected by the steel liner and a 0.3 cm layer of MgO powder. Both walls not used in the test were also protected by the liner and MgO powder packed between the concrete and steel liner. Thermocouple placement in the crucible was only in the sidewalls which were being tested. Thermocouple placement for test 15 is shown in Figure 7. The test had an unprotected magnetite concrete wall and a concrete wall which was protected by two courses of dense siliceous firebrick. The thermocouple placement in the firebrick is shown in Figure 8.

Experimental Test Procedures

Test 14

Test 14 was designed to examine the interaction between molten sodium and unprotected magnetite concrete. The crucible design and test parameters were selected so that the test would be analogous to previous tests in the large-scale test series which used limestone and basalt concretes. The respective test numbers were three² and eleven.¹ Both of the previous tests used shallow sodium pools (15.5 cm and 13.2 cm respectively) and an unprotected crucible cavity.

Test 14 used 123 kg of sodium preheated to 915 K. This resulted in an initial sodium pool depth of 13.5 cm (see Table 1). Immersion heaters rated at 50 kW total power were used to maintain a pool temperature of 825 K. Argon was used as a purge gas. An argon flow rate of 140 liters per minute was maintained throughout the test. Provisions were made to monitor concrete and sodium pool temperatures and penetration rate.

CR1	1 cm FROM CAVITY FLOOR
CR2	1 cm " " "
CR3	2 cm " " "
CR4	2 cm " " "
CR5	3 cm " " "
CR6	3 cm " " "
CR7	4 cm " " "
CR8	6 cm " " "
CR9	19 cm " " "
CR10	2.5 cm FROM BOTTOM OF CRUCIBLE
CR11	1 cm FROM CAVITY WALL
CR12	5.5 cm " " "
CR13	1 cm " " "
CR14	10 cm " " "

Figure 5. Test 14, Crucible Instrumentation.

Figure 6. Test 15, Cavity Construction.

A. PLAINWALL THERMOCOUPLE PLACEMENT

#1-1cm, #2-2cm, #3-4cm, #4-8cm, #5-24cm, #6-37.5cm

B. BRICKWALL THERMOCOUPLE PLACEMENT

#1-1cm #2-2cm, #3-4cm, #4-8cm, #5-24cm, #6-37.5cm

Figure 7. Test 15, Thermocouple Locations in Concrete.

Test 15

Test 15 was designed to examine specific engineering features which are used in FFTF. As shown in Figure 6, the cavity geometry was square. Only two of the sidewalls were used in the test. One sidewall was left as bare concrete. The opposing sidewall was covered with two layers of "Morex", siliceous firebrick which had a density of 2200 kg/m^3 . Two courses, each 7.6 cm thick, were used on this wall (Figure 8). A mild steel liner was then placed into the crucible cavity. In order to simulate flaws in an actual reactor cavity liner the liner had vertical slots 0.6 cm wide by 30.5 cm high in each of the active sidewalls. This allowed the molten sodium to contact the firebrick on one wall and the bare magnetite concrete on the other wall. The region between the firebrick and the liner as well as the bare magnetite wall and the liner was left as open air space. To exclude sodium contact with the remaining two walls and the floor of the cavity, no slots were cut in the liner on those sidewalls or the floor. In addition, MgO powder ("Magox", 80-100 mesh) was packed into the bottom and on the unused sides between the liner and concrete.

The test used 243 kg of sodium preheated to a temperature of 970 K. Immersion heaters rated at 50 kW total power were used to maintain a sodium pool temperature of 825 K. An argon purge rate of 140 liters per minute was maintained throughout the test. The standard provisions were made to monitor temperatures in the sodium pool and concrete, gas generation, and penetration rate in the bare concrete wall using ultrasonic techniques. In addition, aerosol collection devices were included in an attempt to measure aerosol concentration and particle size distribution. Test 15 was the first large-scale test in the basalt-limestone-magnetite test series to attempt aerosol characterization.

In the characterization of aerosols the following information is desired: (1) rate of aerosol production; (2) particle size distribution; (3) aerodynamic behavior including size, shape, and effective density; and (4) chemical composition. All of these items are desired as a function of time during the test since the sodium/concrete reaction apparently goes through energetic and unenergetic phases which would probably produce different amounts and types of aerosols.² Three techniques were incorporated in Test 15: light beam attenuation, inertial cascade impactors, and a wide spectrum particle size sampler. Aerosol concentration can be measured by the light beam attenuation technique. Particle size distribution information can be obtained by the inertial cascade impactors. Aerodynamic behavior and

chemical compositions can be obtained from the material collected on the wide spectrum particle size sampler.

The concentration of aerosol in the gas vented from the top hat was monitored by an opacity, or light beam attenuation, technique. In this technique, the relative intensity of a light beam passing through the aerosol laden gas is related to aerosol concentration. A schematic diagram of the device is shown in Figure 9. The light source was a 6.3 V, 0.35 A incandescent light bulb and the detector was a PIN 10 photodiode. Both the light source and detector were housed in grafoil (Union Carbide sheet graphite) lined chambers. The collimated light beam entered and exited the sample chamber through 0.475 cm diameter holes. The light path length was 3.8 cm. Vanes were used at the entrance of the sample chamber in an attempt to reduce aerosol sedimentation on both the light source and the detector.

Both the concentration detector and the inertial cascade impactors were mounted in a sedimentation volume in the gas vent line immediately outside the test chamber (Figure 10). The distance between the top hat and sedimentation volume was three meters. The concentration detector was fixed to the gas vent line entering the sedimentation volume so that the gas flow was perpendicular to the light beam.

Aerosol samples for determining the aerosol particle size distribution and for calibrating the aerosol concentration detector were collected with two Meteorology Research Incorporated Model 1503 inertial cascade impactors.⁶ These impactors have seven stages which collect particles in the size ranges shown in Table 4 for the gas flow rates used in Test 15. The listed particle sizes are aerodynamic sizes (the equivalent size of a unit dense spherical particle). The collection plates in the MRI impactors were coated with a thin layer of silicon grease per the manufacturer's specifications. Gas flow through the impactors was maintained at a rate of 19 liters per minute by a Gast airpump (Figure 10). The impactors were mounted horizontally inside the sedimentation volume on stands attached to the base of the volume. The airpump was connected to a remotely actuated four way ball valve that allowed each of the collectors to be operated individually for a selected period of time during the test. Gas drawn through the impactors was filtered and reintroduced into the vent line downstream from the sedimentation volume.

In addition to the aerosol concentration device and the inertial cascade impactors, a wide spectrum particle size sampler was designed and constructed for this test. The purpose of including this sampler in Test 15 was to examine the scope of the problem associated with collecting and characterizing aerosols in the sodium/concrete tests. The

Figure 9. Aerosol Concentration Measurement Apparatus
(Using Light Beam Attenuation).

Figure 10. MRI Inertial Cascade Impactors.

TABLE 4

Particle Sizes Collected on Cascade Impactors

Stage	Size Range Collected (μm)*
1	∞ - 27
2	27 - 12
3	12 - 4.5
4	4.5 - 2.2
5	2.2 - 1.2
6	1.2 - 0.6
7	0.6 - 0.4

*Assuming a gas flow rate of 19 l/m through detector.

Figure 11. Wide Spectrum Particle Size Analyzer,
Apparatus Placement.

Figure 12. Wide Spectrum Particle Size Analyzer, End View
Showing Details of Parallel Plate Sampler.

placement and details of this sampler are shown in Figures 11 and 12. To collect the aerosols in the high temperature environments of the sodium/concrete tests, a simple rugged close-in sampler design was used. The sampler was located 1.6 m from the reaction vessel in a straight piece of five cm diameter pipe. When the ball valve in the pipe was opened, aerosol laden gas could flow through the sampler driven by the overpressure in the top hat. The other end of the sampler was open to the ambient atmosphere. Flow rate in the sampler was monitored by measuring the pressure drop across the sampler with an inclined oil manometer. The sampler (Figure 12) consisted of four straight flow channels which were 30 cm long and had narrow dimensions of 0.63 cm for two of the channels and 0.16 cm for the other two channels. The channels were positioned as shown in Figure 12 with the wide dimension extending horizontally. Channel surfaces were made of glass plates for the top two channels and of thin OFHC copper sheets for the lower two channels. This permitted easy removal of both the glass plates and copper sheets for examining particles optically or with the scanning electron microscope. In order to hold particles larger than several hundred microns, one half of all the collection surfaces were coated with a thin layer of silicon grease similar to the coating on the collection plates in the inertial cascade impactors.

Results and Discussion

Test 14

The charge of 123 kg of sodium was heated to 915 K before being transferred into the crucible. Upon transfer, the charge was quenched to 815 K by the concrete crucible. A minimum temperature of 750 K occurred at approximately three minutes. At five minutes, dense clouds of white smoke began escaping from the test chamber. The smoke cleared 20 minutes later and visual observation of the crucible through a small door in the test chamber showed that there was a vertical crack in the sidewall of the crucible. Flames were issuing from the crack and water was dripping from the bottom apex of the crack. An explosion occurred in the cavity of the test article at 42 minutes. This produced a large amount of smoke and flame and disabled the immersion pool heaters. The test cooled monotonically after the explosion.

Figures 13 and 14 show the time-temperature histories of the thermocouples located in the crucible cavity floor (see Figure 5 for thermocouple locations). Duplicate thermocouples were used at depths of one, two, and three cm. Energetic reactions started early in the test as indicated by audible reports in the test chamber and the thermocouple

Figure 13. Test 14, Pool and Concrete Temperatures. Data from Thermocouples CR1, 3, 5, 7, and 8 as Shown in Figure 5.

Figure 14. Test 14, Pool and Concrete Temperatures. Data from Thermocouples CR2, 4, 6, 7, and 8 as shown in Figure 5.

data. The thermocouples located one cm deep in the concrete (CR1 and CR2) indicated that the sodium/concrete reaction front was passing their location at six minutes. The thermocouples located two cm deep (CR3 and CR4) indicated that the reaction front was passing their location at 10 minutes. The thermocouples located three cm deep in the concrete (CR5 and CR6) apparently did not record energetic reactions of similar magnitude as the thermocouples located closer to the original concrete surface. Thermocouple CR5 (three cm deep, Figure 13) showed some evidence of exothermic reaction occurring at 16 minutes. The temperature rise noted by this thermocouple could have been caused by a lower temperature reaction front at this location or it could have been caused by the heat flux from the exothermic reaction noted by CR3 (two cm deep) between the time period 12 to 15 minutes. The latter interpretation would mean that the actual reaction front was between CR3 and CR5 at this time. The general multi-step shape of the time temperature profiles seen by the thermocouples located two and three cm deep could indicate that the energetic periods of the reaction were discontinuous or pulsing. The frequency of the changes in slopes for these curves would indicate that the reaction was pulsing at a rate of one pulse every three to four minutes. The cause of this postulated pulsation is as yet unknown. The maximum temperature recorded by the thermocouples located in the concrete was 1150 K, approximately 65 K hotter than the maximum pool temperature.

The maximum heat flux into the sodium pool caused by the chemical reactions can be estimated from the time temperature histories of the pool thermocouples shown in Figures 13 and 14. The pool temperature rose most rapidly in the time period between 7 and 10 minutes during which time the pool heaters were not supplying heat to the pool (Figure 15). This maximum rate of temperature rise can be used to estimate the heat flux when the reaction was at its most energetic stage. Ignoring conduction and radiation heat loss from the pool, an approximate equation for estimating the heat generation rate is:

$$Q/A = h\rho C_p \frac{dT}{dt} \quad (1)$$

where Q/A = heat flux (J/m^2-s)

h = pool depth (m)

ρ = sodium density (800 kg/m^3)

Figure 15. Test 14, Pool Heater Power and Pool Temperature for Initial Period of Test.

$$C_p = \text{specific heat of sodium (1295 J/kg-K)}$$

$$\frac{dT}{dt} = \text{rate of pool temperature rise (K/s)}$$

Using this equation, the heat generation rate or heat flux into the sodium pool can be estimated at 1.2×10^5 J/m²-s for Figure 13 and 1.3×10^5 J/m²-s for Figure 14. This calculation ignores heat losses from the pool caused by conduction and radiation as well as heat losses from the reaction into the concrete. Because of the large difference in the thermal conductivities of sodium and concrete, the latter losses are expected to be small. Since these heat losses are not considered in the calculation, the actual value of the heat generation rate during this time period may be somewhat larger than the calculated values. The calculated maximum heat generation rates compare favorably with those seen in other large scale tests of limestone concrete² where heat generation rates were on the order of 3.6×10^5 J/m²-s. The heat generation rates calculated here can only be considered estimates, however, because the exact area of the concrete which was reacting with the sodium is unknown. Posttest analysis of Test 14 showed no penetration in the sidewalls, and even though chemical reactions may have occurred at the sidewalls, the area used in the calculations was the floor area of the crucible cavity.

The response of the pool heater controllers correlated very closely with the thermocouple data shown in Figures 13 and 14. Figure 15 shows that the pool heaters began decreasing in power output at 5.1 minutes and at 5.6 minutes had dropped to zero power because the chemical reactions which were occurring had heated the pool above the set point of 825 K. The pool heaters remained at zero power for the remainder of the test.

The penetration rate was measured using a pulse-echo ultrasonic method. This method has been used with apparent success in several large scale tests with limestone concrete.² The results are plotted in Figure 16. The times when the reaction front passed the 1 cm, 2 cm, and 3 cm thermocouples are also shown in Figure 16. Unfortunately, the ultrasonic data are confusing for this test. Two large reflection pulses were observed at approximately the correct position for the sodium/concrete interface during the test. One of these features apparently moved at a relatively slow ("slow" in Figure 16) pace while the other feature moved at a rapid pace ("fast" in Figure 16) during the time period the data were taken. Because of cracking of the crucible and interfering noise pulses, the ultrasonic data were taken for only the first 15.5 minutes of the test. Since the ultra-

Figure 16. Test 14, Penetration as Measured by Ultrasonic Method and Thermocouple Data.

sonic technique is incapable of differentiating as to the correct peak, no conclusions can be drawn from the data. It is possible, however, that the interface at this particular location was quite uneven and that one portion of the interface was moving faster than the other at the time the data was taken. The maximum penetration rate calculated by thermocouple failure is approximately 2.5 mm/min. This corresponds reasonably well to one of the two features ("fast") measured using the ultrasonic methods in the first ten minutes of the test. The penetration rate calculated from the slower feature observed is approximately 2.0 mm/min. At later times, however, this apparently increased. Considering both the ultrasonic data and the thermocouple data we feel that the maximum penetration rate was recorded correctly by the thermocouples and was about 2.5 mm/min.

Posttest examination showed that all of the sodium had reacted. The reaction debris was a frozen pool with craters scattered randomly over its surface and much loose debris on top (Figure 17). The debris bed was not homogeneous. It had a circular center area of approximately 40 cm diameter which had a hard surface layer five cm thick. Beneath this hard layer the debris bed was loose and friable. The remainder of the debris bed outside of this circular area was hard throughout its depth. The bottom of the debris bed which was in contact with the concrete was a bright rust color (area marked by arrows on Figures 17 and 18). The rust colored region was five cm thick at the cavity sidewall and 0.6 cm thick at the cavity center. The layer immediately above the rust colored region appeared to be spalled concrete. The thickness of this layer varied between 0.5 and 2.5 cm. Rust colored material was also on top of this spalled concrete layer and the color of the debris bed gradually changed from black to gray as the top surface of the debris bed was approached. What appeared to be spalled concrete was observed mixed in the black and gray regions. The debris bed was chipped away and a cross section of the crucible cavity floor was mapped. A schematic cross section is shown in Figure 19. There was no erosion of the sidewalls of the crucible cavity. The maximum erosion of the cavity floor was 11 cm.

Chemical analysis was performed on material taken from the center region of the reaction zone. Analyses were performed on (1) an alcoholic extraction, (2) a water extraction, and (3) a residue from these extractions. The relative proportions by weight of the three parts are 1:1:3. The alcohol extraction was determined to be greater than 90 percent NaOH. The water extraction was also highly alkaline with sodium ion accounting for 55 ± 2 % of the mass dissolved. The residue was largely iron oxides and a small amount of CaO , SiO_2 , and Al_2O_3 . The relative proportions of

Figure 17. Test 14, Reaction Debris, Rust Colored Debris
Noted by Arrows.

Figure 18. Test 14, Cored Reaction Debris, Rust Colored Debris Noted by Arrows.

Figure 19. Test 14, Final Penetration Profile.

TABLE 5

Chemical Analysis of Reaction Debris from Test 14*

	<u>Alcohol Extract</u>	<u>Water Extract</u>	<u>Residue</u>
Mass (%)	20 ± 1	21 ± 1	59 ± 1
Alkalinity (meg/gm)	4.46	4.30	
Fe Content (%)	0.16		
Na Content (%)	>18	12.0	
FeO (%)			13 - 14
Fe ₂ O ₃ (%)			33 - 32
SiO ₂ (%)			2
CaO (%)			4
Al ₂ O ₃ (%)			1
Na ₂ O (%)			2

* All figures are in mass percent of original material except alkalinity which is in milliequivalents per gram of original material.

these oxides in the residue were those expected from the original concrete composition. The full analytical results are presented in Table 5.

The extractions indicate that about half of the sodium in the debris is present as NaOH while the remainder of the sodium is combined with iron to form mixed oxides (ferrites) which are unstable in the presence of excess water. X-ray diffraction analysis of the sample identified only the compound NaFeO_2 .

Test 15

The charge of 243 kg of sodium was preheated to 975 K before being transferred into the crucible cavity. A minimum temperature of 700 K was reached at 30 minutes (Figure 20). The pool returned to its set point temperature of 825 K at 125 minutes. During the reheating period, an exothermic reaction between the sodium and siliceous firebrick started behind the steel liner. At 75 minutes, the temperature at the insert brick interface at the bottom of the crucible cavity (location IB-1) rose to approximately 1000 K (location IB-1 in Figure 21, see Figure 8 for thermocouple locations). At the time these temperature excursions began, the pool temperature was approximately 725 K. Figures 22 and 23 show that similar reactions occurred in the firebrick at higher locations starting at 75 minutes and continuing to approximately 120 minutes. The thermocouple data indicate that the sodium-brick reaction front reached the brick-concrete interface at 210 minutes.

Figures 24 and 25 show the thermocouple data for the concrete from the brick faced and bare concrete wall respectively (see Figure 7 for thermocouple locations). There is little indication that exothermic reactions occurred in the vertical concrete walls since the temperatures came up slowly and uniformly as the test proceeded.

During the test (at approximately five hours) a small amount of sodium was observed to be leaking from cracks which had formed in the crucible. The total amount lost was estimated at less than five kg. After five hours of maintaining the sodium pool temperature at 825 K, the pool heaters were turned off and the experiment was allowed to cool in situ.

The ultrasonic data indicated no erosion of the bare concrete wall.

The gas flow data for Test 15 is shown in Figure 26. The argon flow rate has been subtracted from the data shown so that the information plotted is essentially the hydrogen

Figure 20. Test 15, Sodium Pool and Vapor Space Temperatures.

Figure 21. Test 15, Firebrick Temperatures at Bottom of Crucible Cavity (Locations IB-1, BB-1, and BC-1 on Figure 8).

Figure 22. Test 15, Firebrick Temperatures 11.5 cm above Bottom (Locations IB-2 and BB-2 on Figure 8).

Figure 23. Test 15, Firebrick Temperatures 23 cm above Bottom (Locations IB-3, BR-3, and BC-3 on Figure 8).

Figure 24. Test 15, Brick Faced Concrete Wall Temperatures
(see Figure 7 for Locations).

Figure 25. Test 15, Bare Concrete Wall Temperatures
(see Figure 7 for Locations).

Figure 26. Test 15, H₂ Flow Rates.

flow rate during the test. There is an initial burst of approximately 2000 liters/minute of hydrogen but this drops rapidly to approximately 300 liters/minute for the first half of the test. The hydrogen production rate then gradually falls to very low values for the remainder of the test. The initial spike of hydrogen is a combination of thermal expansion of gases in the test chamber as well as rapid hydrogen production attributed to the molten sodium reacting with water released from the concrete.

Posttest analyses showed extensive cracking of the crucible. The top hat/crucible seal was not intact and gases could have escaped via these pathways. Debris formed by the reaction in the firebrick between 75 and 120 minutes could have caused the cracking. This is probably the reason for the gradual decrease in gas flow noted at about this time. There was only minor erosion (< 1 cm) of the magnetite concrete for both the bare and firebrick protected vertical walls.

Measurement of the aerosol concentration and particle size distribution was attempted in this test. As noted previously, three types of apparatus were used to determine these parameters: light beam attenuation for concentration measurements and commercially available impactors as well as a specially designed wide spectrum particle sampler for particle size distribution. The inertial cascade impactors were also used to calibrate the concentration detector.

The wide spectrum particle analyzer was an in-house designed parallel channel flow detector. This test was the first test in which this sampler was used. The sampler was operated at the start of the test. The results from this detector were disappointing. Unfortunately, the sampling time of 320 s was too long and the sampler was seriously overloaded with aerosol. Because of the massive overloading which occurred, no quantitative information was obtained about particle size distribution or even maximum aerosol concentration during the sampling time. For future tests, a much shorter sampling time, on the order of 60 s or less, will be used for this sort of a particle sampler.

The inertial cascade impactors were used to collect two samples of aerosol during the test. The first impactor was operated for the first 11.8 minutes of the test and the second impactor was operated for 9.8 minutes beginning 225 minutes into the test. The operating history for the impactors is shown in Table 6.

When the samplers were retrieved from the sedimentation volume they were quite wet and prior to analysis of the contents they were dried for two weeks in a desiccator filled

TABLE 6

Operating History of Cascade Impactor Aerosol Samplers

<u>Sampler</u>	<u>Time On (min)</u>	<u>Time Off (min)</u>	<u>Gas Volume Drawn (L)</u>	<u>Weight* Collected ---(g)---</u>	<u>Mean Aerosol Concentration ---(g/L)---</u>
A	0	11.8	224.6	9.015	0.040
B	225.2	235.0	186.5	1.174	0.006

* Weights are in error because the sample was rapidly reacting with the laboratory air.

with calcium sulfate desiccant. The moisture is assumed to have collected during the cooldown period of the test. To avoid possible chemical changes in the aerosol samples, arrangements will be made to retrieve similar samples in future tests as quickly as possible after the test.

Both impactors had been overloaded. The amount of material collected on each stage of the first sampler was so excessive that no meaning could be attached to a particle size distribution calculation. The total weight collected by the first sampler was, however, used to calibrate the aerosol concentration detector. The second sampler was not so seriously overloaded. The weights found on each stage in this sampler are listed in Table 7. These data were used to establish a particle size distribution for the aerosol. Cumulative mass weighted probability plots of the distribution are shown in Figure 27. Figure 27 includes the use of both logarithmic and linear horizontal axes for the particle diameter. On each of these respective axes a log normal distribution or normal distribution would be a straight line. The experimental distribution measured by the second sampler is not well represented by either of these distributions. The data does show, however, that the mean particle size of the distribution is between 16 and 19 microns and that the distribution is sharply peaked about this mean.

The size distribution data shown here should be used cautiously. Possible sources of error include overloading of the collector which could interfere with the proper apportioning of the aerosol among the collector stages, and chemical reactions between the aerosol and moist air which occurred between the times the samples were taken and the time they were analyzed. Both of these sources would lead to over-representation of the larger particle sizes in the distribution.

The aerosol collected by the samplers was dull gray in color and ignited when dropped into water. The bulk of the aerosol was most probably sodium particulate. Some of the aerosol was white and did not react vigorously in water but did dissolve to some extent. This material was most likely sodium oxide or sodium carbonate. It was not determined whether the white aerosol was present when the aerosol samples were collected during the test or whether it is an artifact produced after the test by reaction of the aerosol with air during the cooldown period.

The output of the aerosol concentration detector as a ratio of the observed light intensity to the initial light intensity is shown in Figure 28. At the conclusion of the test, the light intensity at zero aerosol concentration was found to have fallen by 50 %. Posttest inspection of the

TABLE 7

Particle Size Distribution From Second Aerosol Sampler

<u>Stage</u>	<u>Weight*</u> <u>(g)</u>	<u>Weight</u> <u>(%)</u>	<u>Cumulative</u> <u>Weight</u> <u>(%)</u>	<u>Particle</u> <u>Size</u> <u>(μm)</u>
1	0.09943	8.47	8.47	27
2	0.77211	65.78	74.25	12
3	0.19005	16.87	91.12	4.5
4	0.05901	5.03	96.15	2.2
5	0.02496	2.13	98.28	1.2
6	0.01764	1.50	99.78	0.6
7	0.00250	0.21	-	0.4
Total	1.17370	-	-	-

*Weights may be in error because the sample was rapidly reacting with the laboratory air.

Figure 27. Test 15, Cumulative Probability Plot of Aerosol Particle Size Distribution.

Figure 28. Test 15, Intensity Ratios for Aerosol Concentration Detector.

detector showed that material had condensed on both the light source and detector. This condensation does not invalidate the calibration of the detector during the times aerosols were being sampled but does reduce the confidence in aerosol concentration data at times between the aerosol samplings.

The aerosol detection system is based on Beer's law:

$$-\ln (I(t)/I_0) = c(t)\epsilon l \quad (2)$$

where

$I(t)$ = observed light intensity at time t

I_0 = light intensity when no aerosol
is present in the light path

$c(t)$ = aerosol concentration at time t

ϵ = extinction coefficient

l = path length

If a sampler drawing a gas at the volumetric rate V collects a sample of weight W during the time interval t_0 to t_f the product ϵl is given by

$$\epsilon l = - \frac{V}{W} \int_{t_0}^{t_f} \ln (I(t)/I_0) dt \quad (3)$$

If condensation on the light detector and light source is assumed to be caused by a time independent concentration, c_0 in the light path then

$$W = - \frac{V}{\epsilon l} \int_{t_0}^{t_f} \ln(I(t)/I_0) dt - Vc_0(t_f - t_0) \quad (4)$$

By assuming that ϵl is a constant, and that condensation on the detector and source had not occurred when the first aerosol sample was taken but was complete when the second sample was taken, the sampler weights may be used to deter-

Figure 29. Test 15, Aerosol Concentrat a.

mine $\epsilon_1 = 17.24$ and $c_0 = 0.071$. Using these values for ϵ_1 and c_0 and the assumption that aerosol condensation on the light source and detector occurred linearly from time 14 minutes to 100 minutes, the data in Figure 28 were converted to the concentration data shown in Figure 29.

At the start of the test, aerosol concentration rises quickly to a maximum of 60 g/m^3 . Concentration falls to a local minimum of about 35 g/m^3 at 30 minutes, then begins to rise to a broad maximum of 40 g/m^3 . A series of sharp bursts in aerosol concentration occurred at 90 minutes and after 125 minutes aerosol concentration falls to a low value near 10 g/m^3 . The sharp bursts that occurred during the time period 90 to 125 minutes correlate well with the temperature excursions noted on the insert-brick and brick-brick thermocouples as shown in Figures 21, 22, and 23.

The concentration of aerosol as shown in Figure 29 is accurate only during those time intervals during which aerosol samples were being taken (0 to 11.8 minutes and 225 to 235 minutes). Elsewhere the results must be considered qualitative because of the assumptions necessary to correct for aerosol condensation on the light source and detector. A strong correspondence exists, however, among the gas flow data (Figure 26), the temperature excursions noted by the thermocouples in the firebrick (Figures 21, 22, and 23), and the aerosol concentration (Figure 29).

The crucible was removed from the test chamber for posttest analysis five days after the test. One centimeter of water covered the test chamber floor. The crucible was extensively cracked both radially and circumferentially as shown schematically in Figures 30 and 31. The reaction products that had built up from the sodium-firebrick interaction had distorted the steel liner inwards towards the cavity of the crucible (Figure 32). The tensile forces in the concrete caused by the expansion of the debris had apparently broken open the sidewall of the crucible. A smaller amount of liner distortion also occurred on the bare wall side of the cavity. The reinforcing bar in the crucible was broken in many places and at the fracture points necking down was noted, which indicates tensile failure. A pool of unreacted sodium 16 cm deep remained in the bottom of the cavity. This amounts to approximately 83 kg of sodium which had not been consumed in the test. The crucible and liner were dismantled. Less than one cm of penetration had occurred in the bare concrete wall of the test. Ultrasonic measurements during the test on the bare concrete wall had indicated that there was no penetration. This correlated well with the posttest analysis.

Figure 30. Test 15, Posttest Elevation View of Cracking Pattern in Crucible (A-D are Eyebolt Sockets Welded to Rebar, Shaded Areas Represent Large Open Cracks).

Figure 31. Test 15, Posttest Top View of Cracking Pattern in Crucible (Crack Widths ~ 0.3 cm).

**Figure 32. Test 15, Photograph of Lined Firebrick Wall
Showing Deformation of Liner.**

Conclusions

The two large scale tests with magnetite concrete demonstrated that an energetic reaction can occur between molten sodium and magnetite concrete. This reaction is capable of producing temperatures as high as 1150 K in the reaction zone. The maximum calculated heat generation rate of $1.3 \times 10^5 \text{ J/m}^2\text{-s}$ is somewhat lower than those seen for limestone concrete, however, it represents a significant amount of energy which can be added to the sodium pool. The maximum penetration rate observed in the tests was on the order of 2.5 mm/min. This rate is essentially the same as rates measured for other types of concrete. The test using the unprotected magnetite concrete showed that the penetration rate is rapid at first and decreases during the test and that it may be a pulsating or discontinuous type of penetration. No sidewall (vertical wall) attack was observed in either test. A maximum of 11 cm downward maximum penetration was observed in Test 14 and all of the sodium was consumed. The experimenters do not know whether the penetration into the floor would have continued had there been more sodium available.

Clearly, siliceous firebrick affords no protection for the concrete. The firebrick was attacked by the molten sodium in a fashion similar to that for basalt concrete. The reaction products which build up from the reaction between molten sodium and the firebrick were capable of extensively deforming the steel liner as well as destroying the concrete crucible and breaking reinforcing bar. Obviously, the forces built up by the reaction products are quite large and are perhaps of concern in reactor safety.

These tests show that magnetite concrete is not inert to molten sodium. The chemical reactions which occur, the heat produced, and the penetration rates are similar to those observed for other types of structural concrete. Precautions, therefore, should be incorporated in reactor designs to preclude the contact of molten sodium and magnetite concrete.

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