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COVER GAS IMPURITIES IN
SODIUM-COOLED REACTORS:
LITERATURE SURVEY

H. P. Maffei

August 1968

AEC RESEARCH &
DEVELOPMENT REPORT

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SODIUM-COOLED REACTORS: LITERATURE SURVEY

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August 1968

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COVER GAS IMPURITIES IN
SODIUM-COOLED REACTORS: LITERATURE SURVEY

H. P. Maffei

INTRODUCTION

A literature survey was conducted to determine what impurities have been found in the sodium cover gases of various sodium-cooled reactors. The emphasis of this survey was to determine the impurity concentration levels observed, the rate impurity levels increased during reactor operation, and what damage, if any, to reactor components may have been caused by the various impurities. Information of this nature will be of benefit in designing the FFTF cover gas system and in orienting R & D work on FFTF cover gas systems.

SUMMARY AND CONCLUSION

The amount of information found on impurity levels of cover gases for sodium cooled reactors is very limited. In fact, for some sodium reactor systems, there appears to be no published analysis of cover gases for impurities. In the survey, no data were found which correlated any reactor damage to cover gas impurities. Data have been found from laboratory tests which show the possible effect of nitrogen cover gas contamination on stainless steels.

SURVEY OF REACTOR SITES: UNITED STATES

LAMPRE

LAMPRE^(1,2) used helium as a cover gas. The sodium was operated in a temperature range of 320 to 930 °F. The makeup helium contained <1 ppm H₂O, ≈ 1 ppm O₂, ≈ 7 ppm N₂, and ≈ 0.5 ppm H₂. As total impurities in the helium cover gas approached 100 ppm, the gas was replaced by flushing.

The references state that typical amounts of hydrogen and methane were observed in the cover gas at startup but returned to nominal concentrations after a short period of operation (concentrations not given). A continuous buildup of nitrogen was observed, as summarized in Table I.

TABLE I. *N₂ Concentration Increase Observed
In the LAMPRE Reactor*

<u>Rate of Sodium Flow at 475 °C, gal/min</u>	<u>ppm/hr</u>	<u>cm³ N₂(stp)/hr</u>
24	5 ± 1	0.4 ± 0.1
122	12 ± 2	1.0 ± 0.2
153	30 ± 10	2.6 ± 0.9

The source of the nitrogen buildup has not been determined. The authors feel that the increase may have been caused by nitrogen diffusing into the system through the 227 ft² of 316 SS surface which was in contact with the sodium.

There are some measurements of fission product activity in the cover gas. Figure 1 shows the normal ²⁴Na activity buildup in the cover gas. Figure 2 demonstrates the rise in cover gas activity due to a fuel capsule leak.

SRE

The SRE^(3,4) reactor used helium as the primary cover gas in the neutron flux. Nitrogen cover gas was used in the remainder of the installation. The sodium coolant temperature was maintained between 500 and 950 °F.

There appears to be no published data of impurity levels in the cover gases other than for radioactive species. Hart⁽³⁾ reports that all of the ¹³³Xe and ⁸⁵Kr released from the fuels was found in the cover gas, but none was detected in the sodium. In an in-pile experiment reported by Kunkel,⁽¹¹⁾ the retention of fission products in 500 °F sodium was determined. The results are summarized in Table II.

Redrawn from Reference 1.

FIGURE 1. Normal Activity Buildup in Cover Gas of the LAMPRE Reactor

Redrawn from Reference 1.

FIGURE 2. Activity Buildup in LAMPRE Cover Gas System Due to Fuel Capsule Leak

TABLE II. *Retention of Fission Products in Sodium at 500 °F in the SRE*

<u>Isotope</u>	<u>Gas Activity, μC</u>	<u>Ratio of Cover Gas Activity to Na Activity</u>
^{131}I	10^{-3}	$<2 \times 10^{-5}$
Ba- ^{140}La	1.8×10^{-3}	10^{-6}
Zr- ^{95}Nb	0	0
^{133}Xe	1.29×10^{-3}	∞ (^{133}Xe not detected in the sodium)

HALLAM NUCLEAR POWER FACILITY⁽⁵⁻⁸⁾

The Hallam reactor used helium at 0.25 psig as a cover gas. The temperature range of the sodium coolant was 607 to 945 °F.

There appears to be no published data on analysis of impurities in the cover gas for this reactor. However, the importance of cover gas analysis is borne out by a failure of three Zircaloy-2 control rod thimbles. The failed thimbles were found to be hydrided below the sodium. Monitoring the cover gas for hydrogen could possibly have suggested the necessity of protective measures to prevent this failure.

Following the replacement of the Zircaloy-2 thimbles with stainless steel, Perret⁽⁶⁾ states "that the hydrogen in the thimble (helium) gas should not exceed 700 ppm." This is in order not to hydride the Zircaloy process tubes beyond 150 ppm over a 10 yr period. The author's calculations take into consideration the diffusion of hydrogen through the stainless steel thimbles and into the sodium coolant.

FERMI

Fermi^(5,9,10) uses argon as an inert cover gas. The sodium coolant is operated at a temperature range of 500 to 800 °F.

The reactor was filled with sodium on November 30, 1960, and maintained at 500 °F through May 1, 1961. It was then heated to 1000 °F for a period of 10 days, maintained at that temperature for 7 days, and then cooled. During the temperature cycle, the offgases from graphite in the reactor combined with the sodium to form free carbon and sodium carbonate. This resulted in the introduction of carbon and oxygen impurities into the sodium as well as the formation of a surface layer of solid reaction products on the sodium surface. The solids layer was analyzed to contain 20.6% C, 37.6% Na_2CO_3 , and 31.7% NaOH.

Cover gas analysis during the 1000 °F operation showed high percentages of CO_2 and O_2 (level not given). Subsequent outgassing tests on graphite under conditions simulating the 1000 °C operation showed hydrogen to be present in the form of methane. Water was also detected.

Typical analysis of the argon cover gas during subsequent normal operations were as follows:

H_2	0-10 ppm
O_2	10
CH_4	0-10
CO	0-50
N_2	3000-4000

Cover gas hydrogen reached a peak concentration of 120 ppm during April of 1966. The hydrogen concentration was found to be correlated with sodium temperature as follows:

- 1) An increase or decrease in sodium temperature resulted in a comparable increase or decrease in the hydrogen concentration.
- 2) Hydrogen concentration decreased with time when the sodium temperature was held constant after a heatup.

The authors feel that "the change in hydrogen concentration during cooldown or at constant temperature appears to be a function of the initial hydrogen concentration, i.e., the higher the initial concentration, the greater the rate of change."⁽¹⁰⁾

Nitrogen concentrations in the argon cover gas have shown a normal range of 1000 to 5000 ppm with a peak of 9000 ppm. When the cover gas is purged, the nitrogen level drops, but then builds up again at a rate of 20 to 40 ppm/hr. The authors state that, "There has been no obvious correlation of the nitrogen buildup with any plant operating condition. Attempts to define the source(s) of nitrogen have not been successful."⁽¹⁰⁾ The authors also show that an extrapolation of the measured nitrogen concentration with time indicates buildup rates of 5 to 10 ppm/hr for systems operating in the 1000 to 1200 °F range.

Occasional oxygen peaks in the 20 to 30 ppm range also have not been explained.

Following the fuel meltdown in the Fermi reactor, the cover gas was purged into a 2500 ft³ tank and two 1000 cm³ samples of gas were taken for analysis. The analysis showed that the cover gas contained 45 Ci of ¹³³Xe and ⁸⁵Kr.

It is possible that the nitrogen buildup in the argon cover gas may have occurred through back diffusion of air into the cover gas. It has been theoretically calculated that air can diffuse into a system against a pressure differential through cracks or holes in nozzles, closures, etc.⁽¹¹⁾ The author showed that the rate of nitrogen buildup by diffusion against a pressure gradient (and consequently a flow of gas) can be expressed by the following relationship:

$$\text{Rate of } N_2 \text{ buildup in ppm/day} = \frac{X^{\circ} N_2 [(\text{leak rate/day}) / \text{total inert gas volume}] \times 10^6}{\left[\exp \left(\frac{\Delta P R^2 g_c}{8 \mu D} \right) - 1 \right]}$$

where,

$X_{N_2}^c$ = mole fraction of N_2 outside the crack

ΔP = pressure differential across crack

R = average effective radius of crack

μ = viscosity of argon at 900 °F, 0.045 cp

D = coefficient of diffusion for the binary system,

assume N_2 in argon $\approx 1 \frac{\text{cm}^2}{\text{sec}}$ at 900 °F

g_c = gravitational constant

With the above relationship, the author calculated various nitrogen buildup rates (Table III) in argon cover gas assuming the following conditions:

- 1) Total volume of inert gas in the reactor vessel 150 ft³
- 2) Rate of leakage of inert gas 0.5 ft³/day
- 3) Length of crack or hole 0.5 ft
- 4) Average effective diameter of cracks 0.2 to 1 mil
- 5) Pressure differential 0.25 to 1 psi

TABLE III. Rate of N_2 Buildup in PPM/Day

ΔP , psi	Radius of Crack, mil		
	0.1	0.25	0.5
0.25	7,880	520	0.04
0.5	3,360	74	6×10^{-7}
1.0	1,210	2	

EBR-II

EBR-II uses argon as a primary cover gas. The sodium coolant is operated at a temperature of 700 to 900 °F.

An analysis of the impurities in the primary argon cover gas showed hydrogen concentrations below 20 ppm. Nitrogen ranged from 0.3 to 1.7% by volume with an average of 1.0%. The secondary cover gas system shows concentrations of 1000 ppm H_2 -He and

500 ppm N_2 . After evacuating the refilling the secondary cover gas system with argon, the H_2 -He content was reduced to <20 ppm.

SEFOR PLANT

Argon will be used in the SEFOR plant⁽⁵⁾ as the primary cover gas at 20 psig. Oxygen is to be maintained below 10 ppm by means of a NaK bubbler. Nitrogen cover gas is to be used in the reactor cavity and the primary sodium equipment areas. The only mention of cover gas analysis by the author states that the nitrogen system is to be monitored for ^{24}Na and the oxygen content of the nitrogen system will be maintained below 5%.

SURVEY OF REACTOR SITES: FOREIGN

UKAEA - DOUNREAY

The Dounreay reactor⁽¹³⁻¹⁵⁾ started operation with a nitrogen cover gas system. The sodium coolant temperature was maintained between 392 and 662 °F. Although cover gas impurities are not reported, an incidence of excessive corrosion of niobium cans is mentioned. The problem appeared to be caused by high sodium temperatures and excessive oxygen in the sodium. No further problems were anticipated so long as the sodium temperature was maintained at a maximum of 480 °F and the sodium oxygen concentration was less than 8 ppm.

J. L. Phillips stated that "A blanket gas (nitrogen) specification of maximum oxygen plus moisture content of 20 ppm was adopted.⁽¹⁴⁾ At levels much above this (amounts not specified) solid skins were formed on the metal and these were very difficult to redissolve." The author also showed that during the course of experiments in the reactor, vanadium and niobium samples were nitrided. The vanadium samples picked up nitrogen to the extent of 4000 to 6000 ppm while the niobium samples picked up 450 ppm of nitrogen. The author felt that the decision to change from nitrogen to argon cover gas in the near future (August 1962) should reduce or eliminate

the nitriding problem. The fact that nitriding did occur in the Dounreay facility demonstrates that nitriding could also be possible in FFTF. Partial pressures of nitrogen in FFTF primary cover gases could lead to nitriding of stainless steel components. The FFTF operating temperatures will be much higher than those of Dounreay. The coolant temperatures for Dounreay were 446 °F inlet and 622 °F outlet (mean).⁽¹⁵⁾

It was found that with the use of vented fuels, ^{133}Xe and ^{135}Xe were always present in the argon cover gas during power operation. The activity levels were 1×10^{10} dpm/liter and 3.7×10^9 dpm/liter for ^{133}Xe and ^{135}Xe respectively, with an associated argon activity of 1 or 2×10^7 dpm/liter. These values varied by a factor of 100 depending on the sample point chosen.

UKAEA - PROTOTYPE FAST REACTOR

The U.K. Prototype Fast Reactor⁽⁵⁾ will employ argon as the primary cover gas. The sodium coolant temperature will be maintained at 1040 to 1085 °F at the core outlet.

A. G. Frame states that the possible use of vented fuels in the Prototype Fast Reactor presents some difficult problems in controlling and removing rare gas isotopes from the argon cover gas. A plant is being designed to remove the rare gas isotopes from the blanket gas. Certain assumptions have been made in designing such a plant. It has been assumed that no retention of krypton and xenon will occur in the fuel itself but the fuel element plenum allows a delay time of approximately 100 hr. The delay time will permit the plant to be designed for the longer lived isotopes only.

The blanket gas cleanup system will pass a portion of the argon gas through activated charcoal beds and then return the gas to the reactor. Because of differences in adsorption characteristics in the charcoal bed, the krypton and xenon will be delayed with respect to argon in passage through the bed.

The author thus feels that by suitable selection of argon flow rates, charcoal volumes, and operating temperatures, most of the active isotopes will decay in the charcoal beds rather than in the gas blanket.

The author states that "An argon recirculation rate of 300 ft³ NTP/min has been selected as the appropriate value to retain the required activity in the gas blanket."⁽⁵⁾ There are no values mentioned for charcoal bed volumes or operating temperatures. A flow diagram of the system is shown in Figure 3.

USSR - BR-5

The BR-5 facility⁽¹⁶⁾ used argon as a cover gas and the sodium coolant was operated at 805 to 930 °F.

The reactor was started up with purified argon as a cover gas. The argon contained <1 ppm O₂ and <0.5 mg/l of moisture.

EFFECTS OF NITROGEN COVER GAS ON AUSTENITIC STAINLESS STEELS

Several reports have been issued which describe the effect of pure nitrogen covered sodium on austenitic stainless steels. Although nitrogen cover gas over sodium will not be used in FFTF, these reports are still of value. The data in these reports give some insight of what may happen to sodium covered stainless steel components in FFTF when nitrogen is present in the cover gas in concentrations of 5,000 to 10,000 ppm. They also show that additional R & D work should be undertaken along these lines.

The reports are summarized below:

- 1) E. G. Brush⁽¹⁷⁾ has found that Type 347 SS is readily nitrided in 900 to 1100 °F sodium having a pure nitrogen cover gas.
- 2) E. E. Baldwin⁽¹⁸⁾ reports that Type 304 SS does not nitride in an 850 °F sodium system through which pure nitrogen gas is bubbled.

1. Primary Tank
2. Condenser
3. Na K Pump
4. Na K Storage Vessel
5. Stack
6. Air Cooler
7. Air Circulator
8. Condenser/Freeze Unit
9. Cold Gas Refrigerator
10. Na Storage Vessel
11. Gas Circulator
12. Gas Circulator
13. Fresh Argon Supply
14. H. P. Gas Storage Vessel
15. H. P. Compressor
16. Filter Bed
17. Gas Circulator
18. Gas Storage
19. Regenerative Heat Exchanger
20. Regenerative Heat Exchanger
21. Delay Bed

FIGURE 3. Gas Blanket Clean Up Plant (UKAEA - Prototype Fast Reactor (Reference 5))

- 3) J. J. Gill⁽¹⁹⁾ has demonstrated that Type 304 SS exposed to nitrogen covered sodium at 1000 °F will nitride. The greatest attack by nitrogen was found to occur slightly below the sodium nitrogen interface. His data is summarized in Table IV.

TABLE IV. *Depth of Nitride Formation in the Vicinity of a Sodium - Nitrogen Interface*

<u>Exposure Time, hr</u>	<u>Sodium Interface, mil</u>	<u>3 in. Below Interface, mil</u>	<u>Nitrogen Side of Interface, mil</u>	<u>3 in. Above Interface, mil</u>
660	0.9	0.1	None	None
1140	1.5	0.1	0.1	0.1
1400	2.0	0.3	--	0.4

- 4) G. C. Wheeler has shown that Type 347 SS will form a nitride case in nitrogen-covered static sodium in temperatures of 800 to 1100 °F.

In a sodium test using a flow rate of 7 ft/sec, it was found that the nitride case was removed resulting in a metal loss from the specimens. Following a 12 months exposure in 800 °F sodium, the coupons showed a loss of 0.3 mil of metal as compared to a loss of 4.8 mils for specimens exposed to 1000°F sodium. (Figures 4 and 5.)

Oxygen or water vapor would form an oxide layer on the stainless steel surface and act as a barrier to nitriding.

PREVENTION OF NITRIDING BY NITROGEN COVER GAS IN EQUIPMENT CELLS

The following reports investigated the oxidation method of nitride prevention. D. J. Keller⁽²¹⁾ reports that "The nitriding reaction is inhibited when small amounts of oxygen are present in a nitrogen atmosphere. Some nitrogen diffuses into the metal but nitrides do not form. The resulting oxidation

FIGURE 4. Nitriding of 347 SS in Static Sodium With Nitrogen Cover Gas (Reference 20)

FIGURE 5. Nitriding of 347 SST in Flowing Sodium With Nitrogen Cover Gas (Reference 20)

attack is much less severe than the nitriding attack." The optimum oxygen concentration was found to be 0.5 vol% at temperatures of 1500 to 1650 °F. The data are shown in Table V.

B. E. Farrel's⁽²²⁾ data demonstrates that exposure to air or N_2 -0.5% O_2 gas for 5000 hr at 900 °F did not change the microstructure of 304 SS specimens. A slight oxidation did occur. However, exposure to pure nitrogen for the same test conditions caused distinct precipitation along grain boundaries. Type 347 SS showed a thin oxide film on the surface after 5000 hr at 1300 °F in N_2 -0.5% O_2 gas. No oxide or metallurgical change was detected on 347 SS samples exposed to pure nitrogen for 5000 hr at 1300 °F.

TABLE V. Maximum Depth of Oxidation of Various Ferrous Alloys Exposed for 100 hr in Nitrogen Containing Small Amounts of Oxidizing Gases* (Reference 21)

Alloy	Maximum Depth of Penetration After 100 hr in Nitrogen Atmosphere Having Addition Shown, mils											
	0.07 vol% H ₂ O			0.2 vol% H ₂ O	0.05 vol% Oxygen		0.1 vol% Oxygen			0.5 vol% Oxygen		
	1500 °F	1650 °F	1800 °F	at 1650 °F	1650 °F	1800 °F	1500 °F	1650 °F	1800 °F	1500 °F	1650 °F	1800 °F
Type 316 SS	**	0.5	1.6	--	0.2	1.0	0.05	--	2.0	0.1	0.15	--
Type 318 SS	0.06	0.06	6.0	0.15	0.05	0.2	0.05	0.05	0.26	0.06	0.05	0.15
Type 347 SS	--	0.05	5.0	--	0.6	0.4	0.06	--	0.3	0.06	0.2	--
Timken 16-15-6	0.3	0.1	1.3	0.5	0.15	0.9	0.1	0.3	1.0	0.2	0.6	0.9
Timken 16-25-6	0.3	0.3	0.6	0.3	0.3	0.5	0.15	0.3	0.5	0.1	0.4	0.6

* Specimen surface was ground to 240-grit finish.

** A dash indicates that the material was not tested.

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