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ABSTRACT

This is an interim report on the modifications which are being made to an existing sodium loop so that a new method of removing hydrogen from sodium can be evaluated in a flowing sodium system.

Some preliminary results on the performance of this type of cold trap alternative in a static sodium system are reported.

Some tentative reactor design parameters for this type of getter device are presented based on the static test results. The life of such a unit is calculated to be substantially greater than that of a cold trap of equal volume.

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I. INTRODUCTION

The efficient removal of hydrogen (and tritium) from sodium is essential to the operation of a Liquid Metal Fast Breeder Reactor (LMFBR). Removal is important in the secondary loop to maintain a low base line concentration to allow the early detection of a water-to-sodium leak in the steam generator system. It is also essential in the primary loop for tritium control. (Hydrogen and tritium behave in a similar manner.) Excess hydrogen in either loop quickly diffuses to the other loop through the intermediate heat exchanger.

Hydrogen trapping from sodium is presently accomplished with a cold trap. Many designs have been developed and evaluated, beginning with the sodium cooled submarine reactor program.⁽¹⁾ Originally it was thought that cold traps had the primary function of oxygen removal, but it is now known that the main load is generally hydrogen (and tritium in the primary circuit). Cold traps are not removal devices, since they only trap impurities.

Despite considerable study, little progress has been made in cold trap design over the last 15 years. Estimates of the life of the present design of secondary traps in the Clinch River reactor is about six months, a serious operating limitation. In addition to poor performance, cold traps are expensive to buy, maintain and operate. As a consequence of the above drawbacks, alternatives for sodium purification deserve serious consideration.

II. COLD TRAP ALTERNATIVES

Alternatives to cold traps which have been proposed to date have not been successful mainly because they were designed to remove oxygen, not hydrogen. It is now known that a stainless steel system will remove oxygen from sodium without a cold trap being used. The cold trap load is primarily hydrogen and tritium in an operating reactor.

This report concerns a preliminary laboratory feasibility evaluation and preparations for a loop evaluation of a new cold trap alternative which was specifically designed to remove hydrogen isotopes from sodium.

To help evaluate this new alternative method of hydrogen isotope removal, a brief review of the behavior of hydrogen in sodium and some of the principles of hydrogen gettering are discussed in Sections III and IV. Sources of hydrogen in a sodium cooled reactor are discussed in Section V.

III. HYDROGEN IN SODIUM

The equilibrium hydrogen pressure over sodium is given by

$$P_{H_2} = 0.204 C_S \quad [1]$$

where

P is hydrogen pressure, torr, C_S is hydrogen concentration in sodium in parts per million (PPM) by weight.⁽²⁾ There is a negligible effect of temperature in the sodium system so this equation is valid over all reactor operating temperatures.

A cold trap operates by cooling the liquid sodium to a temperature where the sodium becomes saturated with hydrogen, at which temperature any excess hydrogen precipitates as sodium hydride. Thus, the level of purity attainable by a cold trap is the concentration corresponding to hydrogen solubility at the cold trap operating temperature. The solubility of hydrogen in sodium is given by the expression

$$\text{Log } C_S = -3180T^{-1} + 6.52 \quad [2]$$

where

T = sodium temperature, K.

Extensive work⁽³⁾ has shown that the hydrogen concentration attained by a well-designed cold trap in an operating reactor is between 100 and 150 ppb (0.15 ppm) and in this report, in making comparisons, the average hydrogen equilibrium pressure corresponding to a good cold trap, is taken as 1×10^{-3} torr. (1.3×10^{-1} Pa)

IV. HYDROGEN REMOVAL BY GETTERING

Metals such as titanium, zirconium and vanadium are known to have the ability to absorb large quantities of hydrogen. This effect is known as "gettering". The rate of absorption is known to depend upon the diffusion coefficient and hydrogen solubility. The behavior of hydrogen in such metals has been extensively investigated. On a theoretical basis, they should be able to absorb hydrogen from sodium so as to achieve a concentration as low, or even lower than that achieved by a cold trap. Except for laboratory investigations, no metal has been commercially successful in suitably purifying sodium. The new gettering method described in this report includes two basic improvements not previously employed:

1. Enhanced solubility by alloying
2. Enhanced permeability by surface modification

The following expression describes hydrogen solubility in the alloy discussed in this report:

$$\text{Log } (C / \sqrt{P}) = 0.14 + \frac{3110}{T} \quad [3]$$

where

- T = absolute temperature, K⁰
 C = hydrogen concentration in metal, ppm
 P = hydrogen pressure, torr

Hydrogen diffusion for a typical alloy is given by:

$$D = 1.58 \times 10^{-3} \exp - \frac{5140}{RT} \quad [4]$$

R = gas constant (2 cal/mole)

Hydrogen permeability, the quantity of hydrogen diffusing through a metal of a given thickness and area, is proportional to the product of the diffusion coefficient and the solubility at the stated temperature and pressure conditions.

When gettering hydrogen from sodium, the alloy permeability dictates the surface area required, and the solubility determines the weight of alloy required to absorb the hydrogen. The equilibrium pressure at a given hydrogen concentration in the alloy must be lower than the equilibrium pressure of hydrogen in sodium at the purity level desired.

A few examples will illustrate how the getter alloy will perform in a reactor.

At a hydrogen pressure of 1×10^{-3} torr, the hydrogen concentration in sodium is 0.15 ppm. However, the hydrogen concentration in the alloy is 8400 ppm at 600F. In a static system, one pound of the alloy could reduce the concentration of a hydrogen in 10,000 lbs of sodium originally containing one ppm down to 0.15 ppm.

As another example, if a new batch alloy were to be placed in sodium at 600F, using a quantity of alloy greatly in excess of that required to absorb the hydrogen, the hydrogen concentration in the sodium could be reduced to only two parts per billion.

Practically, a weight and surface area is determined from the permeability and solubility relations to provide enough surface to absorb hydrogen at its rate of entry into the reactor and enough metal to produce a life of several years.

No uncoated metal or alloy will actually perform according to its calculated potential for hydrogen removal. The explanation for this is that they also have a great affinity for oxygen and form an oxide film faster than they absorb hydrogen. Small amounts of oxygen are always present in sodium, enough to produce an oxide film. This film is an effective barrier to hydrogen permeation, thus destroying the fast gettering potential of the metal.

It has been demonstrated that this problem is solved by coating the alloy discussed in this report with a 0.5 μ m thick film of a metal which does not form an oxide in sodium.

This composite material does actually achieve the theoretical potential of getter material for sodium purification, in contrast to prior failures using the gettering approach.

The application of the coating lowers the permeation rate of hydrogen into the alloy. This is inevitable, since all desirable coatings have a permeation rate much lower than that of the selected alloy. By keeping the protective coating extremely thin, this effect is minimized. A coating thickness of 0.5 μ m was chosen as a thickness giving adequate sodium corrosion resistance combined with minimum resistance to hydrogen permeation.

V. SOURCES OF HYDROGEN IN LMFBRS

In the steam generator of LMFBRS, a substantial amount of hydrogen is generated by the corrosion of the water side of the tubes:

This hydrogen diffuses through the tube wall into the sodium. Diffusion is essentially complete and rapid since the sodium wets the tubes and removes the normal oxide barrier to diffusion.

This hydrogen flux affects plant design in three ways; (A) Cold trap design and operation, (B) Water leak detection (which is based on hydrogen detection) and, (C) tritium control.

The amount of hydrogen likely to enter a reactor from steam generator corrosion has been studied by Roy and Rodgers⁽⁴⁾. They found very good agreement between the results found for several reactors and recommend the use of the following average values for the 2 1/2 Cr - 1 Mo alloy commonly used in LMFBRS steam generators:

Evaporator, steady state flux

$$2 \times 10^{-11} \text{ g hydrogen cm}^{-2}\text{s}^{-1}$$

Superheater, steady state flux

$$1.5 \times 10^{-11} \text{ g hydrogen cm}^{-2}\text{s}^{-1}$$

Initial flux (first 2000 hours for both)

$$8 \times 10^{-11} \text{ g hydrogen cm}^{-2}\text{s}^{-1}$$

The initial rate is experienced when returning a generator to service after each cleaning operation; about every three years for an evaporator.

The initial design of the cold traps for the Clinch River ⁽⁵⁾ reactor was based on hydrogen flux values about an order of magnitude lower than the values given by Roy and Rodgers. If the latter values are correct, the cold traps for the reactor could require replacement after six months of full power operation.

Other sources of reactor hydrogen have not been given enough attention to permit a quantitative analysis of their effects. It is obvious that water leaks in the steam generator will be a hydrogen source and could require high rates of removal for short time periods. Fuel elements and components always introduce hydrogen into a reactor when they first contact the sodium. Hydrogen is evolved until all the absorbed water on their surfaces is reacted with the sodium. The water contained in the make-up cover gas is another source of hydrogen.

Tritium is generated in an LMFBR by fission of a number of actinides and by neutron activation of lithium and boron. A large part of this tritium will enter the sodium coolant and will be partially removed by a conventional cold trap. Generally, the efficiency of tritium removal will depend on the hydrogen flux when a cold trap is used, but good quantitative data on its removal is not available.

VI. STATIC SODIUM TEST - EQUIPMENT

In order to evaluate the effectiveness of the special gettering type alloy, a static hydrogen removal test was run in sodium, using the following equipment and materials:

1. Heated sodium container
2. Hydrogen measuring system
3. System for adding hydrogen to the sodium container
4. Sputter coated alloy

Equipment items 1, 2 and 3 were combined into a sodium container which could be heated to 1000F, attached to a manometer type hydrogen addition system and an ion pump for measuring hydrogen removal. The hydrogen addition was by diffusion through the walls of a closed coil of nickel tubing. The ion pump was attached to a side flange and isolated from the sodium by a 10-mil thick nickel diaphragm. Hydrogen in the sodium diffused through the diaphragm and the rate of diffusion was measured by the ion pump current, which is directly proportional to the hydrogen flux. A line sketch of the system is shown in Figure 1.

Sputter coating was performed by an outside vendor without significant difficulty.

VII. STATIC SODIUM TEST - PROCEDURES

The sodium container was filled from an existing sodium loop to above the side flange to which the ion pump was attached. The sodium entered at the container bottom, and the level was adjusted with a sodium level gage. An outlet line was also provided at the container bottom so that the system could be flushed out before use. The filling lines were then valved off and frozen. Heating was by external resistance heaters, using a controller accurate to $\pm 5F$.

The hydrogen addition system was based upon the use of the gas laws with pressure manometers to determine the amount of hydrogen added. The determination of the volume of all connector lines and the nickel diffusion tube in the sodium was done before sodium filling, using a known standard volume sphere of 217.8 std. cm³ to determine the volume of the rest of the system. Suitable temperature corrections were applied when operating with high temperature sodium. By being able to accurately measure the amount of hydrogen added to the sodium, the ion pump could be calibrated or recalibrated as desired.

The ion pump system was essentially the same as that used to develop the water leak detection system now in use at EBR-II and elsewhere. It consisted of a nominal 11-liter-per-second Perkin-Elmer ion pump with a power supply with the lowest current scale of 0-20 micro-amperes.

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Figure 1. Equipment - Static Sodium Test

The problem of "activation", discussed in the referenced report, was handled by making a long run at about one part per million hydrogen in the sodium until the pump stabilized. Activation studies showed that the speed of the pump increased more than two times, but not the extremes reported by Argonne, (see Figure 2).

The actual test procedures were extremely simple. Hydrogen was added at constant sodium temperature until a hydrogen concentration in the sodium of between 1 and 2 ppm was achieved, based on the increased ion current. The manometers were read before and after addition, and the exact amount added calculated from gas laws. When the hydrogen had diffused uniformly into the sodium, as indicated by a constant ion pump current, the ion pump calibration factor in microamperes per ppm (UA/ppm) was calculated. Two pieces of getter foil were attached to a rod, the top vacuum flange was removed in an inert atmosphere, and the test article was inserted below the sodium level. The flange was then closed and the glove bag removed. Two pieces of foil, coated on one side, were used.

The previously calibrated ion pump current was used as the measure of hydrogen removal. The temperature and ion current were continuously monitored during a run. A run was terminated by reading the final ion current and removing the test article.

VIII. STATIC SODIUM TEST - RESULTS

A. Activation of Ion Pump

It was necessary to "activate" the ion pump to achieve stable readings of the hydrogen monitor. The ion pump current after activation was 13 UA/PPM + 4.5 UA base line with hydrogen removal by ion pump, see plots Figure 2.

In order to allow for hydrogen removal by the monitor the following correction was applied:

Correction for ion pump hydrogen removal rate at 1 ppm (maximum value unactivated):

$$\begin{aligned}
 Q &= SP \quad Q - (8.5) (1.4 \times 10^{-6}) = 11.9 \times 10^{-6} \quad [6] \\
 Q &= 11.9 \times 10^{-6} \frac{\text{torr-liter}}{\text{sec}} = 9 \times 10^{-6} \frac{\text{cc}}{\text{sec}} = 0.8 \frac{\text{cc hydrogen}}{\text{day}} = \frac{0.01 \text{ ppm}}{\text{day}} \\
 &= \frac{0.04 \text{ ppm}}{100 \text{ hrs}}
 \end{aligned}$$

B. HYDROGEN REMOVAL BY GETTER FOIL

The alloy sheet 0.010 inch thick, was cut into 1.19 inch squares and sputter-coated on one side only with 0.5 u of barrier metal. The size used was as large as could be inserted in the vacuum flange opening in the top of the sodium container.

Tests were run at 800F and 600F. The foil would be expected to have a much higher absorbtion capacity at 600F than at 800F, since the solubility of hydrogen in the alloy decreases with increasing temperature. As can be seen by Figure 3, the rate of absorbtion for the composite alloy was not improved by higher temperates even though the diffusion coefficient goes up rapidly with the temperature. The potential rate of absorbtion cannot be evaluated in the unstirred sodium container because the rate of hydrogen absorbtion appears to be controlled by the arrival rate of hydrogen at the alloy surface, which, in turn, is controlled by the rate of hydrogen diffusion in sodium. At the temperature of these experiments the diffusion coefficient of hydrogen in sodium is about $1 \times 10^{-5} \text{ cm}^2 \text{ sec}^{-1}$, while that of the alloy is 2 to 5 times higher. The relative permeation rate, the product of diffusion coefficient and solubility is orders of magnitude higher in the alloy compared to sodium.

Since hydrogen solubility in the alloy is proportional to the one-half power of hydrogen equilibrium pressure in the sodium, the rate of removal is expected to decrease as the concentration of hydrogen in sodium is reduced, and this effect is observed.

An inspection of the results shown in Figure 3 shows that the foil is a very effective getter for hydrogen in sodium without any significant temperature effect under the experimental conditions used.

The broken line in Figure 3 shows the correction needed to allow for the removal of hydrogen by the ion pump. The hydrogen concentration was reduced from 1 ppm to 0.1 ppm in about 200 hours, with a ratio of sodium to getter surface area of 362 g cm^{-2} . The calculated weight of metal required to absorb this much hydrogen would be 2.6 gm, indicating the foil was fully saturated at the end of the experiment.

Although the metal was fairly brittle at the end of the experiment, it had not lost enough strength to present any problem in the removal of commercially sized sheets. No spalling at the base metal was observed, although some of the nickel coating spalled. This is believed to be caused by the use of improper sputtering techniques.

IX. LOOP MODIFICATIONS AND GETTER DEVICE DESIGN

The loop which will be used to evaluate the coated getter device is shown in Figure 4. The loop was originally designed to evaluate fuel cladding and will be called the FCTL in this report.

The modifications required to convert this loop to a hydrogen removal evaluation facility included the addition of the following instruments and device:

- Hydrogen addition system
- Hydrogen concentration monitor
- Hydrogen removal device
- Oxygen concentration monitor

The hydrogen addition system is the same as that used in the static sodium test, Fig. 1. It will be attached to the FCTL at the points marked "A" on Figure 4. The hydrogen monitor will also be the same ion pump used in the static test and will be attached at the same point.

FIG-4 LMFBR FUEL CLADDING TEST LOOP - FCTL

The hydrogen removal device will be attached to the FCTL at the points marked "B" in Fig. 4 and the oxygen monitor will be attached at the points marked "C".

Temperature requirement of the various devices will be met by individual heating and control systems.

The loop will be operated at 600F (316C) since this is near the optimum temperature for the getter device. The hydrogen monitor and addition system will be heated to 800F (427C) to obtain a suitable diffusion rate through the nickel addition tube and ion pump membrane. The oxygen monitor will be operated at 1000F (538C) since this is the minimum temperature required for acceptable sensitivity for the electrolytic type monitor.

The getter device will be initially evaluated at 600F (316C) so no heating system will be required for it. The sodium flow through the hydrogen and oxygen monitors which operate at higher temperature will not be great enough to cause temperature excursions in the removal device.

The getter device design is quite simple. It consists of a containment chamber with a flanged top 3 in. I.D. (76 mm) x 22 in. long (559 mm) with 1 in. (25.4 mm) inlet and outlet connections to the sodium loop.

The getter alloy is a 0.009 in. (0.23 mm) thick sheet, 12 in. (305 mm) x 28 in. (711 mm), rolled into a cylindrical shape to fit inside a 3 in. (76 mm) O.D. x 0.065 in. (1.65 mm) wall tube. The tube with the getter alloy is removable from the loop containment through the flanged top of the containment. The getter sheet is wound on a 2 in. (150 mm) O.D. mandrel and 0.065 in. (1.65 mm) thick spacers are provided to insure uniform sodium flow through the three turns of the getter sheet.

The coating on the alloy is 0.5 μ m thick on both sides of the getter alloy sheet. This thickness was chosen as an optimum with a corrosion allowance for about 5 years of 600F (316C) sodium service while providing a minimum barrier to hydrogen permeation.

The alloy thickness used was not optimized. The use of thin sheet was dictated by the selection available from commercial material which could be handled in the sputtering equipment of a qualified coating vendor and which

could also be manually rolled to fit into a simple containment chamber. Some discussion of the principles of reactor getter design for the coated alloy is required to obtain an appropriate perspective of the configuration and volume of a removal unit of reactor size. The basis of the analysis will be the CRBRP reactor, but the design approach used should apply to any reactor. Design calculations are summarized in Table I.

Table I - Calculated Design Values for CRBRP Hydrogen Removal Unit

A. Alloy Properties

1. Tensile strength $\sim$ 135,000 PSI at 600F
2. Density $\sim$ 5.3 g/cm³
3. Hydrogen solubility 600F, 8000 ppm
4. Hydrogen permeation rate 600F with hydrogen equilibrium pressure over sodium of 1×10^{-3} ton $\sim$ 100 cm³ per cm² per hour for a thickness of 1 mm
5. Permeability of coating is 0.02 cm³ per cm² per hour at 600F (this is controlling rate)

B. CRBRP Assumptions

1. Roy's hydrogen entry data are correct and no other hydrogen source is significant.
2. Total hydrogen entry from steam generator is 27 pounds (12.2 kg) per year.
3. Getter unit will be replaced every 5 years.

C. Getter Design Parameters

1. Weight of getter needed $\frac{(27)(5)}{8 \times 10^{-3}} = 16,875$ lbs
2. Getter area needed (coating is rate limiting)

$$\frac{27}{365(5)(24)} = 0.003 \frac{\text{lb}}{\text{hr}} \text{ H}_2 = 1.4 \frac{9}{\text{hr}} = 15,638 \frac{\text{cm}^3}{\text{hr}} \text{ at H}_2$$

$$\frac{15637}{0.02} = 7.8 \times 10^5 \text{ cm}^2 = 843 \text{ ft}^2 \text{ area needed}$$
3. Size of individual getter pieces assume getter plates are 3 ft x 2 ft, then 69 pieces are needed, 23 in each loop
4. Getter plate thickness
 wt. per plate = 227 lb = 0.685 ft³

$$\frac{0.685 \text{ ft}^3}{6 \text{ ft}^2} = 0.114 \text{ ft thick} = 1.4 \text{ inch thick}$$

5. Volume of getter chamber

Assume getter volume is one-third of total chamber volume, then containment chamber in each loop would be $(0.685)(23)(3) = 47 \text{ ft}^3$. (Present design of CBRRP secondary cold traps specify 100 ft^3 volume but these traps have only 6 month life.) Ratio of containment volume required to cold trap equal life thus equals $\frac{1}{26}$.

X. SUMMARY

Cold traps are presently used for removing hydrogen and tritium from sodium in LMFBFR's. The principal source of hydrogen is corrosion of the tubes by water in the steam generator.

Reliable data are now available on hydrogen entry rates into the secondary sodium and on its diffusion into the primary system. Likewise the behavior of tritium is reasonably well understood. As a consequence of the more accurate information on the quantities of hydrogen and tritium which need to be controlled, the accepted design basis of cold traps seem to be inadequate. Although design improvements seem feasible, the principle of cold trapping to remove hydrogen may be inappropriate for use in a modern reactor.

The principle of gettering to remove hydrogen is not new but has never been successfully applied to purify sodium systems because getter materials meeting the requirements of efficiency, cost and long life in reactor service have not been available.

A new, coated getter alloy has been shown to be effective and provided excellent hydrogen removal in small scale static sodium tests. This report describes preparations being made to evaluate a larger scale model of this type of getter in a flowing sodium loop. Some calculated values of the volume and configuration required for reactor size devices indicate that this type of getter is potentially superior to a cold trap for hydrogen removal.

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