Distribution Category:
Magnetic Fusion Energy
(UC-20)

ANL/FPP/TM-167

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DE83 010691

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IRRADIATION AND COMPATIBILITY TESTING
OF Li_2O MATERIALS AT EBR-II

by

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EBR-II Project/Fusion Power Program

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December 1982

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ABSTRACT

A study was made of the neutron-irradiation behavior of ^6Li -enriched Li_2O material in EBR-II. In addition, a stress corrosion study was performed ex-reactor to test compatibility of Li_2O materials with a variety of stainless steels. Results of the irradiation testing showed that tritium and helium retention in the Li_2O ($\sim 89\%$ dense) lessened with neutron exposure. Helium and tritium retention appeared to approach steady-state after $\sim 1\%$ ^6Li burnup. The effect was likely caused by the formation of open porosity in the pellets. The stress corrosion studies, using a 316 stainless steel (Ti-modified) and a 35% Ni alloy, showed that stress does not enhance the corrosion, and that dry Li_2O is not significantly corrosive, the LiOH content producing the corrosive effects. Corrosion, in general, was not severe as a passivation in sealed capsules seemed to occur after a time, greatly reducing corrosion rates.

I. INTRODUCTION

For the past several years, as a pioneering effort and in anticipation of the utilization of EBR-II as a test bed for irradiation of solid tritium-breeding materials, the EBR-II project has run some preliminary testing of Li_2O materials. The tests have been designed to primarily study the compatibility of Li_2O materials with stainless steel alloys and to measure the tritium breeding and release characteristics of enriched Li_2O in the EBR-II environment.

While the testing provides answers to some safety and operational questions for doing such experiments in EBR-II, the novel data gained from these experiments may provide useful knowledge for design of fusion reactor systems. To this end, this report has been written to summarize the results of the tests and to suggest what future testing may be useful in light of these results.

The results presented in this report will cover the in-reactor tritium breeding and release characteristics of the Li_2O material. In-reactor release characteristics at 550°C and also post-irradiation release at various temperatures ($300, 450, 600^\circ\text{C}$) were examined. Measurements of He generation were used to test predictability of ^6Li burnup. Postirradiation capsule cladding metallography showed the in-reactor compatibility characteristics of the cladding and Li_2O material.

Ex-reactor stress corrosion testing identified the relative importance of temperature, stress, and water content in the Li_2O in the corrosive attack on stainless steel containment materials. A detailed study of the stainless steel materials after testing was used to identify the nature of the corrosive attack and the corrosion products formed.

II. MATERIAL COMPATIBILITY

Stainless steel alloys have been proposed for use in fusion reactor designs. However, little is known about the compatibility of stainless steel materials used in LMFBFR reactors with Li_2O compounds. Earlier studies often have not carefully controlled the water content of the Li_2O , and, because similar compounds, such as NaOH , are known to be particularly corrosive to austenitic stainless steels, it was necessary to do a careful study. Moreover, applied stresses are known to frequently enhance corrosive mechanisms and therefore the effect of stress on compatibility was also investigated.

A. Experiment Design and Description

The test was designed to measure the corrosive effects that Li_2O , in a dry form and also doped with water in amounts representing 1 and 6% LiOH , can have on Type 316 stainless steel (Ti-modified), a high nickel alloy, and a ferritic steel (HT-9). Compositions are subsequently listed in Table III.1. In addition, as the ${}^6\text{Li},n$ reaction produces ${}^3\text{H}$, He is also produced which may pressurize reactor components providing stresses which could enhance corrosion. Therefore, pressurized capsules

were designed and built for testing with pressures as high as 1800 psi (12.4 MPa). Temperatures of 500°C and 600°C were used.

The capsule design is shown in Fig. II.1 along with listings of the pressures, LiOH contents, and temperatures used in the experiment. Each capsule contained a tag gas mixture of inert gases (Ne, Kr, Xe, Ar) along with the He pressurizing gas. They were sealed in evacuated stainless steel cylinders and placed in furnaces. If a capsule failed, a pressure rise would be observed in the cylinder and a gas sample would identify the failed capsule. At one-month, three-month, and one-year intervals, a set of test capsules were removed for examination.

The capsule examination consisted first of laser puncturing the capsule plenums to confirm the presence of the desired pressure. The capsules were then sectioned for metallographic examination of the corrosion depth and character. Further tests were done to identify corrosion products and the chemistry of the Li_2O material.

B. Experimental Results

Figure II.2 shows the composite results of the corrosion depth examinations for one-month and three-month exposures. Figures II.3, II.4, II.5, and II.6 show typical optical micrographs. As demonstrated in the photographs, corrosion was very uniform over the capsule wall in areas near to where the Li_2O material had been. This occurred despite the fact that the Li_2O was in the form of large angular pieces.

STRESS CORROSION CAPSULE

Figure II.1 Capsule Design for Ex-Reactor Stress Corrosion Capsule

Figure 11.2. Corrosion Depth as a Function of Stress for the Stress Corrosion Capsules. Arrows indicate where at least half of the corrosion depth was by grain boundary corrosion.

10 μ m

Figure II.3 Optical Micrographs Showing One-Month Corrosion of Ti-mod. 316 and High Ni Alloy at 500°C and 0.01 LiOH.
a) Ti-mod. 316, low stress; b) high stress; c) High Ni alloy, low stress; d) high stress

Figure II.4 Optical Micrographs Showing One-Month Corrosion of Ti-mod. 316 and High Ni Alloy at 600°C and 0.01 L10H.
a) Ti-mod. 316, low stress; b) high stress; c) High Ni alloy, low stress; d) high stress

Figure II.5 Optical Micrographs Showing One-Month Corrosion of Ti-mod. 316 and High Ni Alloy at 500°C and 0.06 LiOH.
a) Ti-mod. 316, low stress; b) high stress; c) High Ni alloy, low stress; d) high stress

10 μ m

Figure II.6 Optical Micrographs Showing One-Month Corrosion of Ti-mod. 316 and High Ni Alloy at 600°C and 0.06 LiOH.
a) Ti-mod. 316, low stress; b) high stress; c) High Ni alloy, low stress; d) high stress

Evidently vapor-phase transport aided in spreading the corroding chemical species on the capsule wall. The corrosion depth was found to be 5% of the 0.015-inch capsule wall at maximum and was not enhanced by stress, despite the fact that most of the 500°C samples showed intergranular corrosion. Often this type of attack is enhanced by stress. After one month the Li_2O material was found to be nearly dry as LiOH had reacted with the capsule walls and the hydrogen had permeated the capsule walls.

Three-month and one-year examinations showed very similar results to those observed at one month. Evidently, dry Li_2O does not react significantly with these materials at the temperatures tested. Figures II.7 and II.8 show the corrosion after one year for the dry and 6% LiOH conditions. As the LiOH content decreased, the corrosion stopped. The HT-9 corrosion was slightly greater than that observed in the austenitic alloys (see Figure II.9). No capsule failed during testing.

Argon-ion milling combined with Auger scanning spectroscopy showed the chemistry of corrosion products. Typical results are shown in Figures II.10 and II.11. The crusty oxide-looking layer on the surface was found to be MLiO_2 , with the M being mostly iron. Transmission electron microscopy of the material confirmed that the crystal structure closely resembled tetragonal FeLiO_2 ; the lattice parameters varied slightly, likely due to the content of other metal atoms in the structure. The gray layers observed in the metallography were found to be enriched in solutes such as Mo and Nb. Evidently as the MLiO_2 product

Figure II.7 Optical Micrographs Showing 12-Month Corrosion of Ti-mod. 316 and High Ni Alloy at 600°C and 0.06 L10H.
a) Ti-mod. 316, low stress; b) high stress; c) High Ni alloy, low stress; d) high stress

Figure II.8 Optical Micrographs Showing 12-Month Corrosion of Ti-mod. 316 and High Ni Alloy at 600 C and Dry Li_2O .
a) Ti-mod. 316, high stress; b) High Ni alloy, high stress

Figure II.9 Optical Micrograph Showing 12-Month Corrosion of Ferritic Alloy HT-9 at 600 C and 0.06 LiOH. Capsule was in high stress condition.

Figure II.10

Auger Spectrometer Sputter Profile of the Inner Surface of a Corroded Ti-mod. 316 Capsule Showing the Composition of the Outermost Corrosion Layer.

Figure II.11

Auger Spectrometer Sputter Profile of the Inner Surface of a Corroded High-Ni Alloy Capsule Showing the Composition of the Outermost Corrosion Layer.

formed its solubility for these elements was low and they were rejected into the adjoining matrix. Such a corrosion mechanism could tend to slow the corrosion rate as these unreactive solutes build up in the sublayer.

C. Conclusions and Summary

The stainless steel alloys used in this study were chosen as representative of materials which have had extensive data bases developed for use in LMFBR systems. Alloy design has allowed these materials to be particularly resistant to irradiation creep, swelling, and degradation of mechanical properties. It is therefore not necessary to review their merit for use in irradiation environments. In fact, some of these same alloy types have been adapted for possible use in fusion reactor systems; for example, the Ti-modified 316 PCA material. The study was therefore confined to examination of the compatibility of these alloys with Li_2O solid breeder material.

The testing results showed that:

1. Stress, temperature, and Li_2O - LiOH composition conditions covered in this test do not cause significant cladding corrosion over a one-year period.
2. Stress does not enhance corrosion.
3. Dry Li_2O does not cause significant corrosion of the austenitic stainless steels tested.
4. Corrosion which does occur is likely aided by some form of short distance, vapor-phase transport. The resulting corrosion product is of the type MLiO_2 .

5. The two ferritic alloy (HT-9) capsules, which were included only in conditions of highest LiOH content, stress, and time, likewise did not fail at either 500°C or 600°C. The HT-9 samples, however, did show the greatest attack as nearly 10% of the wall depth showed evidence of corrosion after one year at 600°C.

In-reactor testing has shown (see Section III) that radiation does not enhance the corrosion process. The results do show, however, that care must be taken to provide dry Li_2O as a starting material; the LiOH content is directly responsible for the amount of corrosion which does occur.

III. IRRADIATION PERFORMANCE

A. Experiment Description

1. Scope and Objectives

The objective of the experiment was to test the in-reactor performance of capsules containing 85 to 89% dense Li_2O (93% enriched with ^6Li). Issues addressed were compatibility of the oxide with several stainless steel alloys as well as the tritium generation properties of the Li_2O in regards to production, retention, and permeation of the tritium through the stainless steel capsule walls.

Alloys investigated were a Ti-mod. 316 and a high (35%) Ni alloy similar to IN706. The compositions of these alloys appear in Table III.1.

The irradiation environment was provided by the EBR-II reactor using the existing stainless steel creep experiment X147, whose core region temperature is $\sim 550^\circ\text{C}$ (Li_2O pellet centerline temperatures are predicted to be $600\text{--}625^\circ\text{C}$). To use the X147 vehicle, special elements were designed to closely match the geometry of the current 37 creep elements. The 39.5-inch elements each contain four test capsules. A drawing of a test capsule is shown in Fig. III.1. Four of these full-length elements were irradiated. Two full-length elements used 35% Ni alloy encapsulation and two were encapsulated in Ti-mod. 316. Each of the smaller capsules which comprise the longer vehicle element were half-filled with Li_2O pellets. Each X147 creep element contains a 9-inch Ta rod to produce the 550°C Na temperature at core midplane. The

TEST CAPSULE DESIGN

Capsule Pos.	T Clad °C	T _{max} Li ₂ O, °C	ϕ/ϕ_{max} ($E > 0.1$ MeV)
1	557	606	0.55
2	554	624	0.89
3	548	625	1.0
4	538	608	0.86

Figure III.1 Single-Capsule Design Used in the In-Reactor Li_2O Experiment

predicted coolant temperature profile is shown in Fig. III.2. The element environment with respect to creep capsules is shown in Fig. III.3.

2. Irradiation Conditions

Irradiation of the four test elements in X147N began in EBR-II run 113. After two reactor runs (113 and 114), X147 was removed and two of the elements removed for examination. X147 was then reconstituted (X147P) and placed back in EBR-II for four additional reactor runs (116-119) of exposure. This was completed in July 1982.

TABLE III.1 ALLOY COMPOSITIONS (Wt %)

<u>Alloy</u>	<u>C</u>	<u>Ni</u>	<u>Cr</u>	<u>Mo</u>	<u>Mn</u>	<u>Si</u>	<u>Ti</u>	<u>Nb</u>	<u>Al</u>	<u>W</u>	<u>V</u>	<u>Fe</u>
Ti-mod. 316	0.04	15	14.5	1.5	2.3	1.2	0.25	---	0.3	---	---	Bal.
High Ni Alloy	0.03	34	12	0.04	0.3	0.5	1.8	2.9	0.3	---	---	Bal.
HT-9*	0.9	0.5	12.7	0.6	0.2	0.5	---	---	---	0.2	0.3	Bal.

*HT-9 was not included in in-reactor studies, only in the stress-corrosion experiment.

B. Capsule Description, Heat Rating, Component Compositions, and Predicted Tritium/Helium Generation

This section will show the makeup of the experimental capsules with regard to tritium production capabilities.

FIGURE III.2 - Operating Temperature (Cladding) Profile of the In-Reactor Test Elements; Test Capsules are Located In and Near the Core Region.

Figure III.3 Test Element Environment in the X147 Experiment.

1. Weight of Sample (Li₂O)

a. Volume of Li₂O Capsule

Each capsule had a total internal free space of ~ 3.5-inches (8.9 cm) with an I.D. of 0.190-inch (0.483 cm). Of this, 1.25-inches (3.18 cm) was filled with Li₂O pellets, ~ 89% dense. This results in:

$$\text{Capsule Free Space} = 1.627 \text{ cm}^3$$

$$\text{Li}_2\text{O Volume} = 0.581 \text{ cm}^3 = 5.81 \times 10^{-7} \text{ m}^3$$

$$\text{Plenum Volume} = 1.046 \text{ cm}^3$$

b. Weight of Li₂O/Capsule

$$\text{Wgt. Li}_2\text{O} = (2.0 \text{ g/cm}^3) (0.85) (0.581 \text{ cm}^3) = 0.988 \text{ g}$$

c. Weight of ⁶Li/Capsule (93% Enrichment)

$$\begin{aligned} \text{Wgt. } ^6\text{Li} &= (12 \text{ } ^6\text{Li}/28 \text{ Li}_2\text{O}) (0.93) (0.988 \text{ g Li}_2\text{O}) \\ &= 0.394 \text{ g} \end{aligned}$$

2. Irradiation Conditions and ³Li⁶ (n,t) Cross Section

There were four capsules in each element, designated #1, #2, #3, and #4. The following data will represent calculations assuming centerline conditions at each capsule.

<u>Capsule No.</u>	<u>Position with Respect to Core Centerline</u>	<u>T_{Na}, (°C)</u>	<u>(ϕ/[*]ϕ max), (E > 0.1 MeV)</u>
1	+7.75 in.	557	0.552
2	+3.50 in.	554	0.891
3	-0.5 in.	548	1.00
4	-4.7 in.	538	0.860

^{*} ϕ max = $1.562 \times 10^{15} \text{ n/cm}^2 \cdot \text{s}$, total
 = $1.232 \times 10^{15} \text{ n/cm}^2 \cdot \text{s}$, E > 0.1 MeV,
 based upon average of run 107A, row-7 flat positions.

a. ${}^6\text{Li}$ (n,t) Cross Sections

Position 1:

<u>Energy Range, (MeV)</u>	<u>Group Flux, 10^{14} n/cm² · s</u>	<u>$\sigma_{n,t}$</u>	<u>$\Sigma d\sigma$, 10^{14}</u>
0.01-0.1	2.961	1.25	3.701
0.1-0.4	3.121	1.57	4.901
0.4-1.0	1.999	0.55	1.010
1.0-3.0	0.806	0.24	0.193
3.0-6.0	0.168	0.12	0.020

$$\bar{\sigma} = 1.085 \text{ barns} \leftarrow 9.825$$

Position 2:

<u>Energy Range, (MeV)</u>	<u>Group Flux, 10^{14} n/cm² · s</u>	<u>$\sigma_{n,t}$</u>	<u>$\Sigma d\sigma$, 10^{14}</u>
0.01-0.1	3.073	1.25	3.814
0.1-0.4	4.806	1.57	7.546
0.4-1.0	3.748	0.55	2.061
1.0-3.0	1.927	0.24	0.463
3.0-6.0	0.501	0.12	0.060

$$\bar{\sigma} = 0.994 \text{ barns} \leftarrow 13.971$$

Position 3:

<u>Energy Range, (MeV)</u>	<u>Group Flux, 10^{14} n/cm² · s</u>	<u>$\sigma_{n,t}$</u>	<u>$\Sigma d\sigma$, 10^{14}</u>
0.01-0.1	3.287	1.25	4.109
0.1-0.4	5.308	1.57	8.333
0.4-1.0	4.24	0.55	2.333
1.0-3.0	2.200	0.24	0.528
3.0-6.0	0.571	0.12	0.069

$$\bar{\sigma} = 0.985 \text{ barns} \leftarrow 15.372$$

Position 4:

<u>Energy Range, (MeV)</u>	<u>Group Flux, 10^{14} n/cm² · s</u>	<u>$\sigma_{n,t}$</u>	<u>$\Sigma d\sigma$, 10^{14}</u>
0.01-0.1	3.205	1.25	4.006
0.1-0.4	4.701	1.57	7.380
0.4-1.0	3.597	0.55	1.979
1.0-3.0	1.829	0.24	0.439
3.0-6.0	0.472	0.12	0.057

$$\bar{\sigma} = 1.004 \text{ barns} \leftarrow 13.861$$

3. Reaction Rate

The reaction rate for ${}_3\text{Li}^6$ (n,t) is given by:

$$W^* = \phi \text{ total } (\bar{\sigma}) (\text{Wgt. } {}^6\text{Li/at. mass } {}^6\text{Li}) (N).$$

At Position #1:

$$W^* = (9.055 \times 10^{18}) (1.085 \times 10^{28}) (0.394/6) (6.024 \times 10^{23}) \text{ reactions/s} = 3.89 \times 10^{13} \text{ reactions/s.}$$

Similarly, at Position #2:

$$W^* = 5.53 \times 10^{13} \text{ reactions/s.}$$

At Position #3:

$$W^* = 6.08 \times 10^{13} \text{ reactions/s.}$$

At Position #4:

$$W^* = 5.48 \times 10^{13} \text{ reactions/s.}$$

4. Heating Rate

The heat rate for a capsule, H, is given by:

$$H = h (W^*) (1/\text{Volume of Li}_2\text{O}),$$

where

$$h = \text{energy/reaction} = 4.78 \times 10^6 \text{ eV/reaction, and}$$

$$W^* = \text{reaction rate.}$$

Therefore,

$$H (\#1) = 4.78 \times 10^6 (3.89 \times 10^{13}) (1.6 \times 10^{-19} \text{ W} \cdot \text{s/eV}) \\ (1/5.81 \times 10^{-7} \text{ m}^3) = 5.12 \times 10^7 \text{ W/m}^3$$

$$H (\#2) = 7.28 \times 10^7 \text{ W/m}^3$$

$$H (\#3) = 8.00 \times 10^7 \text{ W/m}^3, \text{ and}$$

$$H (\#4) = 7.21 \times 10^7 \text{ W/m}^3$$

The heating rates are low and should not significantly perturb sodium temperatures as shown in the next section. Note: Heating rates contributed from gamma-heating of the Li_2O have been found to be less than 5% of that contributed by the ${}^6_3\text{Li}$ (n,t) reaction, and are ignored.

5. Li_2O Centerline Temperatures (maximum)

The ΔT between sodium (or cladding) temperature, T_3 , and Li_2O centerline temperature, T_1 , is:

$$T_1 - T_3 = \frac{HR^2}{2} \left[\frac{1}{2k_1} + \frac{\ln(r_1/R)}{k_2} \right],$$

where

H = heating rate (see 4)

R = radius of pellet = 0.085-inch

r_1 = gap between pellet and capsule wall (He-filled) + R
 = 0.005 inches + R

k_1 = Li_2O thermal conductivity
 = $3\text{W/m} \cdot ^\circ\text{C}$ at 80% dense, 700°C , and

k_2 = He conductivity
 = $0.318\text{ W/m } ^\circ\text{C}$.

Therefore,

$$\begin{aligned} (T_1 - T_3) &= 49.3^\circ\text{C}, \text{ \#1} \\ &= 70.1^\circ\text{C}, \text{ \#2} \\ &= 77.1^\circ\text{C}, \text{ \#3} \\ &= 69.5^\circ\text{C}, \text{ \#4} \end{aligned}$$

We assume that since the thermal conductivity of the sodium and stainless steel are much greater than the pellet, 24 W/m °C at 700°C for 316 stainless steel, then the Na temperature is not greatly perturbed. If we assume the Na temperature remains as it currently is, then the maximum centerline Li₂O pellet temperature is:

$$\begin{aligned}T_1 &= 606^\circ\text{C}, \#1 \\&= 624^\circ\text{C}, \#2 \\&= 625^\circ\text{C}, \#3 \\&= 608^\circ\text{C}, \#4.\end{aligned}$$

6. Tritium Production, Helium Pressure

The total tritium produced by the Li₂O is:

$$^3\text{T} = W^* (t)$$

where

$$\begin{aligned}W^* &= \text{reaction rate (see C)} \\t &= \text{time, and} \\t &= 3.78 \times 10^6 \text{ s for 1 reactor run.}\end{aligned}$$

Therefore,

$$\begin{aligned}\text{At Position, \#1, } ^3\text{H} &= 1.47 \times 10^{20} \text{ atoms/run, } 8.82 \times 10^{20} \\&\text{atoms/6 runs} \\ \text{At Position, \#2, } ^3\text{H} &= 2.09 \times 10^{20} \text{ atoms/run, } 1.25 \times 10^{21} \\&\text{atoms/6 runs} \\ \text{At Position, \#3, } ^3\text{H} &= 2.30 \times 10^{20} \text{ atoms/run, } 1.38 \times 10^{21} \\&\text{atoms/6 runs} \\ \text{At Position, \#4, } ^3\text{H} &= 2.07 \times 10^{20} \text{ atoms/run, } 1.24 \times 10^{21} \\&\text{atoms/6 runs}\end{aligned}$$

This means that the maximum pressure in a capsule, assuming full helium release to the capsule plenum, is:

$$P = nRT/V; n = \text{moles He, } V = \text{plenum volume,}$$

$$R = \text{gas constant}$$

Since the number of ^3H atoms produced is equal to the He atoms then we can use the previous results of ^3H production for n .

then,

$$P = {}^3\text{H} \cdot T \cdot (1.30 \times 10^{-22}), \text{ atm.}$$

Using $T = T_{\text{max}}$:

$$\text{Position \#1, } P = 100.8 \text{ atm.} = 10.2 \text{ MPa (1,482 psi)}$$

$$\text{Position \#2, } P = 146.0 \text{ atm.} = 14.8 \text{ MPa (2,114 psi)}$$

$$\text{Position \#3, } P = 161.2 \text{ atm.} = 16.4 \text{ MPa (2,370 psi)}$$

$$\text{Position \#4, } P = 142.2 \text{ atm.} = 14.4 \text{ MPa (2,088 psi)}$$

This translates into a maximum hoop stress of:

$$\text{Position \#1, } \sigma_h = 73.2 \text{ MPa} = 10.6 \text{ ksi}$$

$$\text{Position \#2, } \sigma_h = 106.0 \text{ MPa} = 15.4 \text{ ksi}$$

$$\text{Position \#3, } \sigma_h = 117.2 \text{ MPa} = 17.0 \text{ ksi}$$

$$\text{Position \#4, } \sigma_h = 102.0 \text{ MPa} = 14.8 \text{ ksi}$$

C. Experimental Results

The previous section of this report described the experiment that was irradiated in EBR-II. The following section describes the postirradiation examination of the capsules contained in elements EA-401 and 403

which were irradiated for two reactor runs and capsules from EA-400 and 404 which were irradiated for six reactor runs.

1. Postirradiation Examination of EA-401 and EA-403

Postirradiation examination included laser puncturing of each capsule to determine plenum volume and to collect plenum gas for He and ^3H measurement, sectioning of capsules for metallography and removal of Li_2O pellets, measurement of He and ^3H content of Li_2O pellets, chemical and radiochemical analysis of selected Li_2O pellets and the calculation of He and tritium production and retention characteristics of each capsule. A further test was made to study tritium permeation rates through the walls of the irradiated capsules at 300, 450, and 600°C.

EA-401 and EA-403 were elements comprised each of four sealed capsules containing ~ 1 gram of Li_2O pellets. The pellets are 93% enriched in ^6Li and were fabricated to be 85-90% of theoretical density. EA-401 was built from an experimental γ'/γ strengthened alloy. It is an alloy containing ~ 35% Ni and is similar to commercial alloy Inconel 706. EA-403 was constructed from an austenitic alloy which is essentially a 316 austenitic stainless steel modified with Ti to help void swelling and creep resistance. The exact compositions of the alloys appear in Table III.1. The tubing used was 0.015-inches thick with an I.D. of 0.200-inches and an O.D. of 0.230-inches.

a. Tritium Permeation - Two capsules numbered 1 and 4 from EA-401 and EA-403 were analyzed for tritium release rates at 300, 450, and 600°C. Each capsule tested was inserted into a quartz tube and placed in a tube furnace connected to a tritium train. Each capsule was fitted with a quartz holder to elevate the capsule above the bottom of the quartz tube during heating. Tube furnace heat was controlled with a temperature controller and the temperature was monitored with an independent thermocouple. Argon carrier gas at a flow of 100 cc/min swept released tritium from the tube to a CuO-tube furnace maintained at 600°C where tritium is converted to tritiated water. The converted tritium was removed from the carrier gas by bubbling the gas through water cooled in an ice bath (catch solution). During sampling of released tritium, the capsule was held at the desired temperature for 3 to 4 hours and samples of converted tritium were collected. Sampling times were 30 minutes per sample. Generally, six samples were taken at each temperature. After appropriate dilutions, tritium activity was measured by liquid scintillation counting. Measured tritium release rates for each capsule at each temperature are given in Table III.2. Tritium release rates were constant over the measurement periods, except for EA-403, #4 at 672°C where the last two iterations showed a significant reduction in release.

b. Laser Puncturing - The volume of the capsule plenums were measured and the plenum He and tritium collected for measurement by placing the capsule in a laser-puncturing enclosure connected to an evacuation and sampling system. The volume of the puncturing enclosure

was determined prior to and after puncturing the cladding. The increase in volume was taken as the plenum volume. Gases escaping from the punctured plenum were collected for He and tritium measurement.

c. Capsule Cutting - Capsules were cut into sections to provide samples for metallurgical and chemical analysis and to remove Li_2O pellets. As sections of cladding material and Li_2O pellets were removed, they were placed in a bottle, blanketed with argon and sealed. Selected Li_2O pellets were removed from the hot cells for dissolution and analysis.

d. Chemical and Radiochemical Analysis of Li_2O - Four Li_2O pellets from capsules 2 and 3 from each element were analyzed for elemental impurities, activation products, and elements which may have migrated from the cladding. The results are given in Table III.3.

e. Helium and Tritium Measurement in Li_2O - The helium and tritium content of Li_2O pellets were measured by weighing each pellet, dissolving each pellet in H_2O , capturing the evolved gas, then measuring the tritium in each dissolver solution and the tritium and helium content of the evolved gases. The results of these measurements combined with the results obtained from laser puncturing were used to calculate He and tritium production and retention. The results of these measurements are outlined in Table III.4.

TABLE III.2 TRITIUM RELEASE RATES

<u>Capsule</u>	<u>Temperature (°C)</u>	<u>³H Release Rate (μCi/s)</u>
EA-401, 1	300	8.9×10^{-3}
EA-401, 1	450	2.2×10^{-1}
EA-401, 1	600	16.0
EA-401, 4	300	1.3×10^{-1}
EA-401, 4	450	9.2
EA-401, 4	600	68.9
EA-403, 1	300	2.7×10^{-3}
EA-403, 1	450	2.9×10^{-1}
EA-403, 1	600	9.8
EA-403, 4	385*	1.4
EA-403, 4	518*	78
EA-403, 4	672*	713

*Estimated temperatures; initial temperature readouts were in error.

TABLE III.3 Li₂O PELLET CHEMICAL AND RADIOCHEMICAL ANALYSIS

		<u>Chemical Analysis (ppm)</u>					
		<u>Fe</u>	<u>Ni</u>	<u>Cr</u>	<u>Mo</u>	<u>Zn</u>	<u>Nb</u>
EA-401	#2	120	103	<23	<73	45	<40
	#3	135	128	<26	<83	93	---
EA-403	#2	130	89	<27	<87	44	<46
	#3	106	101	<26	<82	62	---
		<u>Radiochemical Analysis (d/s/gram)</u>					
		<u>⁷Be</u>	<u>⁵⁸Co</u>	<u>⁵⁸Mn</u>	<u>⁶⁰Co</u>	<u>¹⁸²Ta</u>	
EA-401	#2	9.10×10^4	2.81×10^5	4.24×10^4	5.88×10^3	2.07×10^4	
	#3	9.10×10^4	4.53×10^5	7.20×10^4	5.37×10^3	-----	
EA-403	#2	1.20×10^5	6.75×10^4	2.41×10^4	7.10×10^3	5.52×10^3	
	#3	8.55×10^4	8.35×10^4	4.02×10^4	5.92×10^3	1.43×10^4	

TABLE III.4 BURNUP ANALYSIS AND TRITIUM RELEASE FOR CAPSULES EA-401, 403

Sample	Li ₂ O wt. (gm)	He		He cc in Plenum (atoms)	Tot. He(cc) (Tot. atoms)	³ H Ci/gm Li ₂ O (atoms)	³ H μCi in Plenum	³ H atoms He atom X100	³ H, Ci released during heat Experiment (atoms)	Plenum Pressure psia	Plenum Vol. cc
		cc/gm Li ₂ O (atoms)	cc/gm Li ₂ O (atoms)								
401-1	0.2338	9.11 (2.45x10 ²⁰)	3.75 (1.01x10 ²⁰)	12.86 (3.46x10 ²⁰)	1.18 (2.45x10 ¹⁹)	60.6	8.13%	0.18 (3.63x10 ¹⁸)	49.7	1.30	
401-2	0.3101	8.48 (2.28x10 ²⁰)	5.10 (1.37x10 ²⁰)	13.58 (2.65x10 ²⁰)	1.13 (2.35x10 ¹⁹)	6.4	6.4%	--	65.5	1.32	
	0.2749	--	--	--	1.28 (2.66x10 ¹⁹)	--	7.3%	--	--	--	
401-3	0.2587	7.36 (1.98x10 ²⁰)	7.23 (1.94x10 ²⁰)	14.59 (3.92x10 ²⁰)	1.00 (2.08x10 ¹⁹)	27.5	5.3%	--	89.6	1.35	
	0.2415	--	--	--	1.26 (2.62x10 ¹⁹)	--	6.7%	--	--	--	
401-4	0.2345	9.68 (2.0x10 ²⁰)	4.33 (1.16x10 ²⁰)	14.01 (3.76x10 ²⁰)	0.60 (1.25x10 ¹⁹)	3.4	8.40%	0.92 (1.91x10 ¹⁹)	55.9	1.33	
	0.2203	--	--	--	0.83 (1.72x10 ¹⁹)	--	9.70%	--	--	--	
403-1	0.2367	8.48 (2.28x10 ²⁰)	5.06 (1.36x10 ²⁰)	13.54 (3.64x10 ²⁰)	0.95 (1.98x10 ¹⁹)	32.4	6.15%	0.11 (2.28x10 ¹⁸)	63.9	1.34	
403-2	0.1519	8.53 (2.29x10 ²⁰)	4.85 (1.30x10 ²⁰)	13.37 (3.59x10 ²⁰)	1.07 (2.22x10 ¹⁹)	12.3	6.2%	--	61.6	1.34	
	0.2293	--	--	--	1.27 (2.64x10 ¹⁹)	--	7.4%	--	--	--	

TABLE III.4 BURNUP ANALYSIS AND TRITIUM RELEASE FOR CAPSULES EA-401, 403 (contd)

Sample	Li ₂ O wt. (gm)	He cc/gm Li ₂ O (atoms)	He cc in Plenum (atoms)	Tot. He(cc) (Tot. atoms)	³ H Ci/gm Li ₂ O (atoms)	³ H μCi in Plenum	³ H atoms He atom X100	³ H, Ci released during heat Experiment (atoms)	Plenum Pressure psia	Plenum Vol. cc
403-3	0.2526	6.16 (1.65x10 ²⁰)	5.43 (1.46x10 ²⁰)	11.59 (3.11x10 ²⁰)	1.19 (2.47x10 ¹⁹)	6.06x10 ²	7.9%	--	69.4	1.33
	0.1253	--	--	--	1.20 (2.49x10 ¹⁹)	--	8.0%	--	--	--
	0.2525	--	--	--	1.18	--	7.9%	--		
403-4	0.1799	5.86 (1.58x10 ²⁰)	7.07 (1.90x10 ²⁰)	12.93 (3.48x10 ²⁰)	0.72 (1.51x10 ¹⁹)	1.39x10 ⁴	50.0%*	7.67 (1.59x10 ²⁰)	94.9	1.25

*Includes ³He atoms released during heat-release rate experiments.

The total He found in the pellets and plenum of the capsules ranged from 3.1×10^{20} atoms to 3.9×10^{20} atoms. Preirradiation predictions based on the assumed neutron spectrum and flux in row seven of EBR-II, and published ${}^6\text{Li}$ reaction cross-section data indicated that 3.1×10^{20} to 4.6×10^{20} atoms of He would be produced, representing a little over 1 percent burnup of ${}^6\text{Li}$. Results and prediction were in surprisingly close agreement and give credence both to our confidence in future predictive capabilities and in our postirradiation experimental techniques.

It was found that approximately 60-70% of the He produced remained in the pellets. The pellets retained structural integrity shown by the fact that they could be removed intact from the capsules.

Corrosion of the stainless steel containment materials was minimal and no more than observed in the ex-reactor testing. Figure III.4 shows the degree of attack of the Li_2O material on the 35% Ni material. Comparison to the plenum region demonstrates that some attack had occurred but was limited to $\sim 2\%$ of the wall thickness. Metallographic examinations have not yet been completed, but no significantly greater attack is expected in other capsules as very little variation was observed in the ex-reactor tests, not only as a function of material type, but also as a function of temperature.

Tritium retention, or release, results proved to be very puzzling. Most capsules retained 8-9% of the tritium produced.

Fig. III.4-MICROSTRUCTURE OF 35%-Ni CLADDING FOLLOWING IRRADIATION ($\sim 0.8 \times 10^{22}$ N/cm², $E > 0.1$ MeV) AND CONTACT CONCURRENTLY WITH ENRICHED Li₂O PELLETS. PLENUM AND CONTACT REGIONS ARE SHOWN. IRRADIATION TIME WAS ~ 90 DAYS (2 EBR-II CYCLES). CORROSION DEPTH WAS FOUND TO BE ONLY 8 MICRONS (2% OF WALL).

Capsule 4 of element EA-401 retained more than 10%, while capsule 4 of element EA-403 retained 50% of the tritium. Nearly all of the tritium found appeared to be trapped within the pellets.

During transfer, however, the low-flow subassembly tends to overheat due to self-heating of the tantalum material which serves to heat the flowing Na during irradiation. Thermal expansion temperature monitors (TEDs), which record a maximum temperature, showed that temperatures as high as 793°C were reached in the axial position near capsule 4. Higher axial positions were also heated in excess of the 550°C operating temperature, but the overheating was less at higher axial positions. Since subassembly transfer may take several hours, it is likely that some of the retained tritium was lost during this time. High tritium content found in the subassembly wash water support this assumption. Steps were taken in handling the second transfer (EA-400, 404) to minimize these problems. In addition, the capsules remain in the reactor basket before transfer for periods of several days which could result in the loss of ~ 0.1 Ci of ^3H , or $\sim 10\%$ of the retained tritium.

Tritium permeation (release rate) test results provided another key to the puzzle. Note in Table III.2 that the release rates were much higher for capsules 4 from both EA-401 and EA-403 than in sister capsules 1. This supports the finding that more tritium had been retained in these number 4 capsules, although that found in capsule 4 from EA-401 was only slightly higher than in the other capsules.

The most likely cause of the higher retention was operating temperature. The capsule position 4 is positioned below reactor core centerline, and, because the tantalum heats the sodium coolant as it flows upward through the subassembly, this position operates at a lower in-reactor temperature. It is predicted, however, that the temperature is only 20-30°C cooler than that of the other capsules. This indicates that there may be a rather dramatic temperature dependence of the ability for the Li_2O pellets to retain tritium. The results from EA-400 and EA-404, as demonstrated in the next section of this report, support the temperature effect. Future testing may include irradiation at $\sim 400^\circ\text{C}$ in order to assess the full extent to which operating temperature may influence tritium retention.

There was also another, indirect support for a sharply changing correlation between in-reactor temperature and tritium retention. When the Li_2O pellets were dissolved for analysis of He and ^3H contained within them, some of the ^3H was released to the plenum of the gas sampling apparatus. The remaining amount of ^3H was trapped in the water used to dissolve the pellet. It was found that $\sim 17\%$ of the ^3H was released to the plenum of the dissolution flask for pellets removed from capsules 1, 2, and 3. However, for pellets from capsules 4, only 4% of the tritium was released to the plenum. This very likely indicates that the dissolution mechanism is different for the pellets which retained more tritium. Moreover, a variation in dissolution mechanism likely indicates that the phase distribution for tritium containment in the Li_2O pellets has been altered.

2. EA-400 and EA-404

EA-404 was made from Ti-mod. 316 while EA-400 was made from the 35% Ni alloy. Experimental conditions were identical to EA-403 and EA-401 except they were irradiated approximately 3 times longer. Postirradiation examinations on these elements therefore permit examination of Li_2O pellet properties, tritium retention, He retention, etc. as a function of irradiation exposure or burnup of ^6Li .

To date the examination of these capsules has consisted of a burnup analysis, together with a close scrutiny of the He and ^3H retention characteristics. Metallography, designed to measure the degree of corrosion present in the capsule walls, has also been completed. These examinations have been completed on capsules #2 and #4 from each element, thereby saving capsules #1 and #3 fully intact for any proposed future examinations.

a. Tritium Retention and Burnup Analysis

Table III.5 lists data for capsules #2 and #4 from elements EA-400 and EA-404 in a similar fashion to the data listed in Table III.4. Direct comparison of these two tables allows examination of the time and irradiation exposure effects on helium and tritium retention.

Note that the percentage of helium retained in the pellet is much less after the longer exposure. Moreover, note that the

TABLE III.5 BURNUP ANALYSIS AND TRITIUM RELEASE FOR CAPSULES EA-400, 404

<u>Sample</u>	<u>He cc/gm Li₂O (atoms)</u>	<u>He cc in Plenum (atoms)</u>	<u>Tot. He(cc) (Tot. atoms)</u>	<u>³H Ci/gm Li₂O (atoms)</u>	<u>³H μCi in Plenum</u>	<u>³H atoms He atom X100</u>	<u>Plenum Pressure psia</u>	<u>Plenum Vol. cc</u>
400-2	3.92 (1.06x10 ²⁰)	39.2 (1.06x10 ²¹)	1.16x10 ²¹	0.625 (1.47x10 ¹⁹)	39.6	1.3	495	1.29
400-4	9.46 (2.55x10 ²⁰)	32.9 (8.87x10 ²⁰)	1.14x10 ²¹	2.23 (5.25x10 ¹⁹)	74.0	4.6	420	1.28
404-2	4.40 (1.19x10 ²⁰)	40.0 (1.08x10 ²¹)	1.12x10 ²¹	0.70 (1.65x10 ¹⁹)	68.3	1.4	504	1.28
404-4	7.55 (2.03x10 ²⁰)	34.2 (9.20x10 ²⁰)	1.12x10 ²¹	3.08 (7.25x10 ¹⁹)	266.0	6.5	430	1.29

tritium retained in the pellet material is also a smaller percentage of the total produced compared to EA-401 and EA-403.

A plausible explanation for these trends is that the helium helped to open a network of interconnected porosity in the Li_2O which allowed most of the helium produced after that point to be released to the capsule plenum. The rapid release of helium could then perhaps act as a carrier for tritium release.

As seen in the EA-401, 403 results, (Table III.2) the capsules #4 showed a higher degree of tritium retention compared to capsules #2. This same performance was demonstrated again at higher burnup in the EA-400, 404 results. The phenomenon is again likely due to a slightly lower irradiation temperature for capsules #4. Note, however, that the time/exposure-dependent drop in the percentage of tritium retained in the Li_2O pellets (the onset of a steady-state release) occurs at the lower temperature as well, despite the slightly higher retention. If a change in phase is associated with the higher retention at lower temperature, it does little to prevent the release of tritium after a few percent burnup of ^6Li .

Metallography of the Li_2O pellet material will be needed to help sort out the reasons for the high release rates after extended irradiation exposure. Confirmation of whether or not a network of interconnected porosity develops would help to prove or disprove the

existence of an enhanced release mechanism based upon helium acting as a carrier gas.

When dissolving Li_2O pellets in the more highly irradiated condition, it appears that a consistent value of $\sim 6\%$ of the tritium is released to the plenum of the dissolution flask for all pellets. Recall that a much higher amount was released to the plenum for high temperature pellets (#1, #2, #3) after the shorter irradiation exposure.

b. Pellet Swelling

While pellet immersion density or metallography have not yet been done, one can estimate the Li_2O pellet $\Delta V/V_0$ from data shown in Tables III.4 and III.5.

Note that measured capsule plenum volumes have decreased between the original 2-cycle exposure and the 6 cycle exposure. The Li_2O pellets occupy a volume of $\sim 0.6 \text{ cm}^3$ in each capsule. The plenum volume reduction during the final 4 cycles was $\sim 0.045 \text{ cm}^3$. Assuming this ΔV is caused by Li_2O swelling, then the Li_2O swelled by $\sim 7\%$ during the second exposure sequence.

It is likely that this additional swelling is not directly related to He production and retention because approximately the same amount of He was retained in the pellets after the second exposure than the first. Irradiation damage induced swelling or phase transformations are possible causes.

20μm

Figure III.5 Optical Micrographs of Irradiation Capsule Walls (EA-400, 404-2) After $\sim 3.0\%$ ${}^6\text{Li}$ Burnup (2.5×10^{22} n/cm², $E > 0.1$ MeV). Pellet and Plenum Regions are Shown, But the Difference is Less Than Observed for the Shorter Irradiation. Maximum Corrosion is 3% of the Wall. a) Ti-mod. 316, plenum; b) pellet region; c) 35% Ni Alloy, plenum; d) pellet region.

c. Stainless Steel Corrosion

Metallography of the capsule materials after irradiation is depicted in Figure III.5 for the two different materials. The corrosion layer depth is not three times that observed in capsules EA-401 and EA-403 but is very similar in magnitude. This very closely parallels the results of the ex-reactor testing, where a marked decrease in the corrosion rate was seen after a short exposure time. It was concluded that the effect was due to a drying of the Li_2O . This conclusion still seems valid for the stress corrosion experiment, but the in-reactor experiments provide evidence of a second effect which results in the same passivity effect.

The in-reactor exposure results in continuous generation of tritium, so one would expect continued corrosion. The apparent passivation may therefore be attributed to near-surface concentration of certain solutes as observed in the ex-reactor samples. This solute segregation may even be enhanced by radiation-induced solute segregation mechanisms. While the complete answers are not discernible at this time it is apparent that corrosion of these austenitic stainless steel materials by Li_2O - T_2O compounds during tritium breeding and release is not of serious concern to reactor design or maintenance, at least over exposure times of the order of magnitude studied here.

d. Conclusions

(1) EA-400, 404 results have confirmed that burnup predictions made prior to the irradiation were quite accurate. Burnup

measurements after irradiation were based on measuring the total He contents of Li_2O pellets and capsules plenums.

(2) The relatively constant He and ^3H concentrations retained in the Li_2O (4-9 cc He/gm Li_2O , ~ 0.5 -3.0 Ci ^3H /gm Li_2O) indicated a steady-state release behavior after ~ 1 at.% burnup of ^6Li . The He retention behavior with ^6Li burnup appears similar to fission gas retention in nuclear fuels, and therefore may have been caused by the development of open porosity in the pellet microstructure. Pellet metallography or density measurements may confirm this, but further development into experimental techniques is needed to do this accurately. Tritium release increases may then be related to a carrier gas effect, where open porosity and He transport aid in transport of tritium out of the Li_2O material.

(3) ^3H release data for EA-400, 404 confirmed earlier findings showing that the capsules #4, which operated at ~ 520 -530°C (cladding temperature) compared to the 550-560 temperatures of the other capsules, retained significantly more tritium. The apparent temperature effect may be confirmed by a follow-on experiment run at 400°C.

(4) Cladding corrosion was surprisingly small in magnitude when extrapolated from EA-401, 403 results. An apparent surface passivation occurred after short exposure times. This may be related to surface segregation of certain solute species.

ACKNOWLEDGEMENTS

Many Argonne people besides the primary authors took part in these experiments and their help was most appreciated. We would like to especially thank R. M. Arons, L. A. Neimark, R. B. Poeppel, V. K. Sethi (MST); C. E. Johnson (CET); Y. I. Chang (AP); B. G. Carlson, R. I. Martell, R. Villarreal, R. S. Wisner (ANL-W); E. L. Wood (EBR-II).

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