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A FAST NEUTRON FLUX: BEATRIX-II, PHASE I AND II

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IN SITU TRITIUM RECOVERY FROM Li_2O AND Li_2ZrO_3 IRRADIATED IN A
FAST NEUTRON FLUX: BEATRIX-II, PHASE I and II

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ABSTRACT

BEATRIX-II was an in situ tritium recovery experiment designed to characterize the behavior of lithium ceramics irradiated to high burnup in a fast neutron flux. This in situ tritium recovery experiment provided data on the performance of Li_2O and Li_2ZrO_3 under irradiation conditions covering a range of sweep gas compositions and temperatures. The experiment consisted of two separate irradiation cycles which in turn included two vented tritium recovery canisters each. Phase I operated for 300 Effective Full Power Days (EFPD) while Phase II operated for 203 EFPD of irradiation. Both Phase I and II resulted in lithium burnups in excess of 4%.

The tritium recovery behavior of Li_2O and Li_2ZrO_3 was characterized using temperature transients, sweep gas composition changes and reactor power changes to effect changes in the tritium inventory. These parameter changes indicated that increasing temperatures or increasing concentrations of hydrogen in the sweep gas, in general, resulted in a decrease in the tritium inventories in the lithium ceramic. The results of these parameter changes allow comparison not only in the tritium recovery behaviors for Li_2O versus Li_2ZrO_3 but between different Li_2O microstructures.

The high neutron flux level in FFTF resulted in high tritium generation rates which combined with a responsive tritium measurement system allowed detailed observations on the tritium recovery behavior of Li_2O and Li_2ZrO_3 . During the course of the experiment a number of observations were made which did not appear consistent with a simplified view of the tritium recovery behavior of these materials. These observations included small negative tritium recovery peaks preceding the typical primary positive peaks; and, for specific temperature ranges, changes in tritium inventory which were opposite to the characteristic expected changes. This anomalous behavior together with the extensive BEATRIX-II database of tritium recovery results provides an opportunity for a better understanding of the tritium release behavior of these two candidates for a fusion solid breeder blanket.

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INTRODUCTION

The BEATRIX-II irradiation experiment is an in situ tritium recovery experiment to evaluate the tritium release characteristics of fusion ceramic breeder materials and to characterize their stability under fast neutron irradiation to extended burnups. This IEA sponsored experiment was carried out in the Materials Open Test Assembly of FFTF. The participants are Japan, Canada and the U.S. The in situ tritium recovery experiment consisted of two individual in-reactor experimental assemblies identified as Phase I and Phase II that were irradiated for 300 and 203 EFPD, respectively. Each experimental phase included two specimens: a thin annular specimen capable of temperature changes and a larger temperature-gradient specimen. In Phase I both specimens were Li_2O while for Phase II the temperature-change specimen was Li_2O while the temperature-gradient specimen was Li_2ZrO_3 .

The design and operation of these experiments along with the results of the individual experiments have been reported previously in detail[1-8]. The purpose of this presentation is compare the tritium recovery results for Phase I and II and to highlight nontypical behavior. The reason for emphasizing this nontypical behavior is that it reflects details of the tritium release behavior from the solid breeder material and provides an opportunity for a better understanding of the tritium release behavior of these two candidates for a fusion solid breeder blanket.

EXPERIMENT DESCRIPTION

Both Phase I and Phase II included Li_2O ring specimens in a temperature controlled canister. The enrichment in the Phase II specimen was increased to 95% from the 61% enrichment in Phase I to increase lithium burnup rate. Phase I achieved 4.8% burnup in 300 days while Phase II achieved the same burnup in only 203 days. The ring specimens were thin-walled tubes 8.9 cm long with an outer diameter of 1.84 cm and wall thickness of 0.16 and 0.1 cm. The wall thickness of the Phase II specimen was made thinner than the Phase I specimen in an attempt to minimize the temperature gradient across the specimen and also to achieve as low as possible minimum temperature. The temperature is monitored/controlled by thermocouples located on the inner (hotter) surface of the specimen. The minimum achievable temperature for Phase I and II were 550 and 530°C. The maximum temperature was chosen to be approximately 100°C higher than this or 640°C. During irradiation at full power, the temperature difference across this specimens is calculated to be 40 and 20°C. The temperature of the specimen is controlled by changing the thermal conductivity of the gas in the thermal-gap of the canister [1,2]. Temperature changes occur in time periods less than 15 seconds. The sweep gas used for the in situ tritium recovery flows along the inside of the ring specimen from bottom to top.

Although the Phase I and Phase II temperature gradient specimens were very different (i.e., Li_2O pellets and a Li_2ZrO_3 sphere bed), the canister designs were very similar. In both canisters the "solid" specimens had outer temperatures near 440°C with centerline thermocouples to monitor the inner temperatures of 1000 and 1100°C. The sweep gas entered the canister at the bottom and flowed around or, in the case of the sphere bed, through the canister exiting at the top.

Table 1. Description of the Specimens and the Irradiation Conditions for the BEATRIX-II Experiment

Canister Type	Phase I		Phase II	
	Temperature-Change	Temperature-Gradient	Temperature-Change	Temperature-Gradient
Specimen	Li ₂ O	Li ₂ O	Li ₂ O	Li ₂ ZrO ₃
Form	Ring	Solid Pellet	Ring	1 cm Spheres
Weight (g)	11.95	34.31	8.1	29.5
Outer Diam(cm)	1.85	1.70	1.85	1.32
Inner Diam(cm)	1.55	0.27	1.65	0.23
Length(cm)	8.9	8.9	8.9	8.9
⁶ Li Enrichment	61 %	61 %	95 %	85%
Temperature (°C)	550-640	440-1000	530-640	440-1100
Experiment Duration (EFPD)	300	300	203	203
Lithium Burnup	4.8%	4.1%	4.8%	5.2%

RESULTS: TEMPERATURE-CHANGE CANISTERS

For the Phase I and II temperature-change canisters the primary emphasis of the test plan was on the effect and interrelationship of gas composition and temperature on the tritium recovery from Li₂O. Temperature transients were carried out at intervals throughout the experiment to characterize the effect of burnup and irradiation history [4,5,8].

Temperature Transients

In general, temperature transients resulted in positive tritium recovery peaks for temperature increases and negative recovery peaks for decreases. Figure 1 shows typical tritium recovery peaks for a Phase I temperature change series of 640-600-550-600-640-530-640°C in the reference sweep gas of He-0.1% H₂. This series was run during Phase I after 42 EFPD. The transient recovery peaks in this series were double peaks which consisted of an initial HT peak followed by an extended HTO peak.

Figure 3 shows a progression of recovery peaks in a sweep gas of He-0.1% H₂ for a Phase I temperature transient of 550-640°C compared with a 530-640°C transient at the start of Phase II. The Phase I peaks are from a series of temperature transients used to characterize the effect of burnup on the tritium recovery. At the start of the Phase I, the peaks were double peaks similar to those shown in Figure 2. By the end of Phase I, (300 EFPD) the HT peak dominated the total recovery peak. There was some question as to what caused this change. Two explanations proposed were burnup effects in the specimen or a drying out of the system. The occurrence of a predominantly HT peak at the start of Phase II suggested that the effect seen in Phase I was

not due to burnup. Therefore the cause of the change in Phase I may be due to a drying out of the system that continued into Phase II.

Temperature transient series were also carried out during Phase I to characterize the effect of burnup. The tritium recovery peaks for a 530-640°C temperature change in 0.1% H₂ are shown in Figure 3. The first three peaks at 4, 51, and 61 EFPD are similar in shape, as was expected. The last two peaks at 151 and 200 EFPD are preceded by small negative peaks. The peak at 142 EFPD was not a part of a series of temperature transients but was for an isolated 530-640°C temperature transient following an extended period of operation at 530°C in sweep gases of He and 0.01% H₂. This transient recovery peak at 142 EFPD consisted primarily of HTO in contrast to the previous peaks which were primarily HT. The subsequent 151 EFPD peak was taken from a temperature transient series and was appreciably smaller than the peak at 142 EFPD. Although the 151 EFPD peak consisted primarily of HT, it retained the initial negative peak feature.

The effect of sweep gas composition on the tritium recovery behavior was addressed using a sequence of temperature changes in both Phase I and II. Figure 4 is a comparison of the Phase II recovery curves for the temperature change from 530 to 640°C in the three different sweep gases: 0.1% H₂, 0.01% H₂, and He. Decreasing the hydrogen in the sweep gas resulted in much larger tritium recovery peaks. Integration over the peaks was used to determine the respective amount of tritium associated with the three peaks: 0.04, 0.23, and 0.76 Ci. This increasing change in inventory with decreasing hydrogen concentration is consistent with the increasing tritium inventories in the Li₂O specimen found for decreasing hydrogen concentration.

Sweep Gas Composition Effects

Increasing hydrogen concentration in the helium sweep gas resulted in a positive transient tritium recovery peak. Conversely, decreasing hydrogen concentration in the sweep gas resulted in a steep drop in recovery rates followed by a slowly increasing recovery rate. This behavior could be considered to be broad negative peaks that required a longer time to reach equilibrium than in the case of increasing the hydrogen concentrations. Similarly, the faster recovery times observed for increasing hydrogen concentrations to 0.1% H₂ is probably due to a faster return to equilibrium in the higher hydrogen concentration rather than indicating that inventory decreases in the Li₂O occurred more rapidly than inventory decreases.

Sequences of sweep gas composition changes started and ended with 0.1% H₂ sweep gas. Figure 5 compares the gas composition sequence of eight days in He followed by two days in 0.01% H₂ at 640°C for Phase I and II. The behavior in Figure 7 is typical of the tritium recovery behavior observed during sweep gas composition changes. The tritium recovery behavior for Phase II exhibited sharper, smaller tritium recovery peaks than Phase I indicating a faster response time and smaller changes in inventory. Thus the larger grain size and higher density of the Phase II specimen did not appear to increase the residence time of tritium in the specimen.

Reactor Power Changes

Reactor power changes, particularly startup and shutdown, provided an opportunity to study temperature changes in the temperature-change canisters which are below the range of normal operating conditions. Although power changes result in a change in both the neutron flux and tritium generation rate, the dominant parameter change affecting the tritium recovery behavior was the temperature change. In both Phase I and II, reactor startup and shutdown behavior of temperature change canisters containing Li_2O was strongly dependent on the composition of the sweep gas. In a 0.1% H_2 sweep gas, decreasing the power (tritium generation rate/specimen temperature) resulted in a decrease in the tritium recovery rate as expected. However, in a helium sweep gas, the tritium recovery curve during decreasing power contained positive recovery peaks. Figure 6 is the recovered tritium and the temperature for the Phase I temperature-change canister during a shutdown in helium sweep gas. A major recovery peak started at about 470°C and continued to the end of shutdown. Such an observation suggests that a decreasing tritium inventory occurs in the Li_2O specimen with decreasing temperature. The smaller recovery peaks near the start of the shutdown are primarily due to temperature variations that occurred in the canister as the temperature control system tried attempted to maintain the temperature as the neutron heating decreased. In Phase II transient recovery peaks were also observed during shutdown in helium but the peaks, starting at 580 and 450°C, were significantly smaller than the peak observed for Phase I.

RESULTS - TEMPERATURE-GRADIENT CANISTERS

One of the most obvious indicators of the physical stability of the Phase I and Phase II temperature-gradient specimens was the long-term stability of the specimen temperature during irradiation. The primary parameter change in the test plan for these canisters was the sweep gas composition. Although temperature changes were not part of the canister design, reactor startup and shutdown provided opportunities for observing the tritium recovery under changing temperature and tritium generation.

Centerline Temperature

The Phase I Li_2O specimen was operated with in a center temperature near 1000°C. During the first ten days of operation the center temperature dropped approximately 30°C, but after that a slow steady decrease in the temperatures was observe throughout each reactor cycle. Because of the uncertainty in predicting the thermal conductivity of the Li_2ZrO_3 sphere bed the design temperature was in the range from 1000 to 1200°C and the final temperatures were slightly less than 1100°C. For the Phase II temperature change canister the temperatures in the center of the specimen decreased uniformly through out each of the 50 day cycles. The slow steady decrease in temperature during each reactor cycle in Phase I and II was attributed to a decrease in power at the canister position.

Sweep Gas Composition Effects

As was observed for the temperature-change canisters, sweep gas composition, in particular the amount of H_2 in He, was found to have a major effect on the steady-state tritium recovery from both Phase I [6] and Phase II[7] temperature-gradient canisters. Figure 7 is a comparison of the tritium recovery data for 4 days in He for the Phase I canister containing solid Li_2O versus the Phase II canister containing Li_2ZrO_3 spheres. The main difference in the recovery curves is that the integrated areas under the peaks, both the negative peak at the introduction of He sweep gas and the positive peak at the return to He-0.1% H_2 , are smaller for Li_2ZrO_3 . The inventory change for the large positive Li_2O peak is 14.4 Ci, while that for the Li_2ZrO_3 peak is 0.76 Ci. For the Phase I Li_2O centerline temperatures were approximately 100°C less than the Li_2ZrO_3 spherebed. The tritium generation rate was about 3 times larger in the Phase I Li_2O . Thus, although the tritium inventory increases in a helium sweep gas for both ceramics, but the inventory buildup is smaller for the lithium zirconate. Note that a small secondary HTO peak occurred for Li_2O at reintroduction of He-0.1% H_2 , but it is masked by the width of the large HT peak.

Figure 8 is a series of sweep gas composition changes carried out for the Phase II temperature-gradient canister that involved 4 days in He followed by 2 days in 0.01% H_2 . Compared to the recovery rate in 0.1% H_2 the tritium recovery curves indicated a 20% reduction of recovery rate in He gas followed by a recovery rate in 0.01% H_2 gas which was only 8% less. Upon re-introduction of the 0.1% H_2 reference gas, the recovery rate returned to its original value. The tritium recovery behavior during the He to He-0.01% H_2 transition has an unexpected negative peak preceding the normal positive recovery peak. This negative peak is similar to the negative recovery peaks observed for the Phase II temperature-change canister during temperature transients in Figure 3. Sweep gas changes for the Phase I temperature-gradient canister containing Li_2O did not exhibit this type of behavior.

Reactor Shutdown

During reactor startup/shutdown, the tritium recovery behavior of the Phase I temperature-gradient specimen in reference gas (i.e. He-0.1% H_2) was as expected. However, reactor shutdown at the end of Phase I-B under helium sweep gas was found to have a significantly different tritium recovery behavior. Figure 9 is a plot of tritium concentration and centerline temperature for the Phase I temperature-gradient canister during reactor shutdown both in 0.1% H_2 and He. The temperature of the specimen decreased in direct response to the decrease in neutron intensity. The tritium recovery during the shutdown in He-0.1% H_2 decreased steadily as the power was reduced. For a He sweep gas, the behavior was drastically different. As the neutron level decreased, the tritium generation rate and specimen temperature decreased but the recovered tritium concentration increased. For the Phase II temperature-gradient canister the shutdown in a helium sweep gas was similar to the shutdown in He-0.1% H_2 .

In the case of the temperature-change canisters the tritium recovery peaks during shutdown could be related to a specific temperature range in the specimen. The broad range of temperatures in the temperature-gradient specimen make it difficult to determine if the phenomena was specific to a temperature range and, if it is, to identify a temperature range. However, tritium retention results during postirradiation examination indicated that the majority of the tritium inventory is in the outer regions of the specimen where the temperature is less than 540°C. This suggests that the tritium recovery peak for the temperature gradient canister is related to the same temperature range that was responsible for the recovery peak for the Phase I temperature change canister.

DISCUSSION

The tritium recovery behavior for the BEATRIX-II, Phase I and II Li_2O canisters indicated tritium inventories decrease at higher temperatures and or at higher hydrogen concentrations in the sweep gas. Two more-detailed observations common to both Phase I and II invite further discussion. The first was the occurrence of small "negative" peaks preceding the positive recovery peaks that resulted from changes to higher temperature for Li_2O or to higher hydrogen concentration for the Phase II Li_2ZrO_3 canister. The other observation was the reduction in inventory as the temperature was being decreased during shutdown in a helium sweep gas, i.e., "inverted" peaks.

Negative Recovery Peaks

The occurrence of small negative peaks preceding the larger positive peaks after temperature increases was observed in BEATRIX-II, Phase I, the CRITIC-I experiment [9,10] and by Tanaka, et al., [11]. In BEATRIX-II, Phase I these negative peaks were only observed early in the experiment. In CRITIC-I the behavior was observed prior to conditioning at 800 to 850°C and it was proposed that conditioning of the specimen decomposed a surface layer of Li_2CO_3 . Kopasz, et al., [12] have shown that the CRITIC-I behavior could be modeled using a desorption activation energy that varies with surface coverage. Tanaka, et al., [11] found a strong relationship between the occurrence of the negative peaks and water vapor in the recovered sweep gas. Alternatively, Asaoki, et al., [13] have shown a correlation of these negative peaks with the production of F° centers.

In the Phase II temperature-change canister, the occurrence of these negative peaks appeared to have been brought on by the extended operation in He and 0.01% H_2 at the lower temperature of 530°C. Operation at this lower temperature in sweep gases with reduced hydrogen (<0.1% H_2) had a large component of HTO in the tritium recovery peaks resulting from a return to the reference state, i.e., when the hydrogen was increased to 0.1% and the temperature was raised to 640°C. Analogous to the conditioning of the CRITIC-I specimen at 800 to 850°C, this recovery behavior suggests that a "reverse" conditioning of the specimen may have occurred at the lower temperatures in reduced hydrogen sweep gases. This reverse conditioning in the Li_2O specimen may have been associated with a buildup of tritium inventory in the form of dissolved moisture. The occurrence of the large component of HTO in the

recovery peaks during a return to the reference state was consistent with Tanaka, et al., [10] findings of the association of the phenomena with higher moisture levels.

For the Phase II Li_2ZrO_3 canister, the occurrence of negative recovery peaks was observed in connection with sweep gas changes to higher hydrogen concentration, specifically for the change from He to He-0.01% H_2 . This observation is referred to as a negative peak because it has a similar appearance to the negative peaks associated with the temperature transient curves. Negative peaks occurring for sweep gas composition changes have not been reported previously. Since the behavior was not seen for the Phase I, temperature-gradient canister, it would appear as if the phenomena was characteristic of the tritium release behavior of Li_2ZrO_3 and not Li_2O .

Inverted Recovery Peaks

The observation of tritium recovery peaks during shutdown in helium was inverse to the behavior observed during constant power operation. For the Phase I temperature change canister, a major peak was observed starting at about 470°C. The large peak occurring for the Phase I temperature gradient canister appeared at the start of shutdown and it is inferred from the postirradiation examination that the peak is associated with regions of the specimen with temperatures less than 540°C. In Phase II small recovery peaks were seen at 580 and 450°C.

These tritium recovery peaks during shutdown in helium suggest that if temperature transients were carried out in the specific temperature regions associated with these peaks, temperature decreases should result in tritium inventory decreases. The small peaks seen starting at 580°C in the Phase II canister suggest that the effect should occur for temperature changes in the vicinity of this temperature. Such decreases were not seen during the temperature transients carried out in Phase II from 640-590-530°C and the reason may have been because the temperature intervals were too broad and the decreases were masked by the overall increase. The Phase II shutdown peak in the vicinity of 580°C was not a particularly large peak and could easily be overshadowed by a larger temperature drop. However, in Phase I the shutdown peak in the 470-430°C temperature range was much larger and one could expect to see such an "inverse" effect in temperature transients carried out in the lower temperature regions. During the initial part of the CRITIC-I experiment on Li_2O , low temperature transients in the range of 460 to 620°C resulted in inverted peaks[10]. That is, a decrease in temperature resulted in a positive peak in the tritium recovery curve. The CRITIC-I experimental observations included a temperature transient from 540 to 480°C in helium sweep gas that resulted in a positive tritium recovery peak. This behavior is very consistent with the behavior seen in the BEATRIX-II, Phase I and II shutdown curves in helium. As noted above for the reverse peaks, the inverted peaks in the CRITIC-I test occurred in the first part of the test and were not observed later on in the experiment after the 800 to 850°C conditioning. It was proposed that the inverted peaks were associated with the solubility of tritium in a surface layer that was decomposed or eliminated during conditioning.

The 470-430°C temperature range associated with the major recovery peak during shutdown in helium is in the range where the LiOT or dissolved moisture is an important factor in determining the tritium inventory [14, 15] in Li_2O . Because the moisture levels in the BEATRIX-II sweep gas are typically less than 0.1 Pa, the amount of moisture in the specimen is expected to remain below the LiOH solubility limit as the temperature is decreased. If the dissolved moisture within the specimen remained in equilibrium with the helium sweep gas, it is not expected that LiOH dissolution in Li_2O would be a factor in the tritium recovery during shutdown in helium. A previous discussion of the Phase I behavior [5] suggested that the variation of water vapor pressure over LiOT dissolved in Li_2O may have contributed to the shutdown behavior. At the present time the explanation of the inverse peaks is not complete. However, operation in helium and the subsequent buildup of tritium inventory does appear to be associated with a buildup of dissolved moisture in the specimen as suggested by the large HTO peaks observed when the specimen is returned to the reference condition. This buildup of tritium inventory in the Li_2O is one of the primary differences in the specimens operated in helium versus 0.1% H_2 . The occurrence of these inverse peaks during the shutdown in helium and not in 0.1% H_2 suggests that the buildup of tritium inventory during operation in helium, possibly in the form of dissolved moisture, may contribute to this behavior.

CONCLUSIONS

The results of the BEATRIX-II, Phase I and Phase II irradiation experiment have provided an extensive data base on the in situ tritium release characteristics of lithium-ceramic solid breeder materials for lithium burnups as high to 5%. In general, the tritium recovery results indicate higher temperatures and higher hydrogen concentrations in the sweep gas lead to lower tritium inventories in Li_2O . Nontypical behavior observed during temperature transients, sweep gas composition changes and reactor power changes provide opportunities for additional insight into the mechanisms controlling tritium release from these lithium ceramic solid breeders. The temperature-gradient canister provided data for in situ tritium recovery from a Li_2O specimen or a Li_2ZrO_3 sphere bed irradiated under a large thermal gradient typical of fusion blanket designs. The thermal stability and tritium recovery behavior of the BEATRIX-II, Phase I and II canisters provide additional verification of the viability of Li_2O and Li_2ZrO_3 as candidates for a fusion solid blanket.

LIST OF FIGURES

- Figure 1 Tritium recovery peaks for a temperature change series of 640-600-550-600-640-530-640°C in the reference sweep gas of 0.1% H₂
- Figure 2 Progression of tritium recovery peaks for a 550-640°C temperature transient in Phase I versus a 530-640°C temperature transient in Phase II
- Figure 3 Tritium recovery peaks for temperature transient 530-640°C at 4, 51, 61, 142, 151, and 200 EFPD.
- Figure 4 Tritium recovery curves for a temperature change from 530 to 640°C in the three different sweep gases: 0.1%H₂, 0.01%H₂, and He
- Figure 5 Comparison of 640°C tritium recovery curves for the Phase I and II sweep gas composition sequences of eight days in He followed by two days in 0.01% H₂
- Figure 6 Recovered tritium and temperature for the Phase I temperature-change canister in helium sweep gas during a reactor shutdown.
- Figure 7 Comparison of the tritium recovery curves for 4 days in He for the Phase I canister containing solid Li₂O versus the Phase II canister containing Li₂ZrO₃ spheres.
- Figure 8 Recovered tritium during sweep gas composition changes for 4 days in He followed by 2 days in He-0.01%H₂ for the Phase II temperature-gradient canister.
- Figure 9 Tritium concentration and centerline temperature for the phase I temperature-gradient canister during reactor shutdown both in 0.1% H₂ and He.

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Phase I Ring Specimen Temperature-Change Series

94021002.SPW 04/19/94

94012012.SP.W 04/14/94

Phase I Temperature-Change Canister Shutdown in Helium Sweep Gas

BEATRIX-II, Phase I and Phase II Temperature-Gradient Canisters

94041403.SPW 04/14/94

Temperature Gradient Canister (DT-3S-3) 4 days in He, 2 days in He-0.01% H₂

Phase I Temperature-Gradient Canister

Shutdown Behavior in Helium