

Determination of the order of surface reactions in Li₂O

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An analysis of the different surface reactions taking place in Li₂O was performed in order to determine whether adsorption and desorption of tritium are first or second order reactions. Data from BEATRIX-II Phase I and CRITIC-I were used as basis for calculations. It was found that only second order adsorption/desorption on the surface of Li₂O can predict the tritium behavior observed experimentally.

يعتبر اكسيد الليثيوم واحد من المواد المقترحة كمولد للترينسيوم في أغلفة مفاعلات الإنماج، ولذلك من الضروري فهم سلوك التريتيوم المولد بداخل تلك المادة، مع تحديد تأثير كل عملية على ذلك السلوك. كما يجب فهم تفاعلات السطح وتأثيرها على سلوك ومخزون التريتيوم في اكسيد الليثيوم. تم تحليل تفاعلات السطح المختلفة في اكسيد الليثيوم، وذلك لتحديد درجة الإدمصاص والمج ومعرفة إن كانوا من الدرجة الأولى أم الثانية. وتم أخذ بيانات من تجريتي BEATRIX-II و CRITIC-I كأساس للحسابات. أظهرت الحسابات انه فقط تفاعلات الإدمصاص والمج من الدرجة الثانية ممكن أن تتوقع سلوك التريتيوم الظاهر في التجارب.

Keywords: Li₂O, Adsorption, Desorption, Surface processes

1. Introduction

Lithium oxide is considered to be one of the candidate solid breeder materials for blankets in D-T fusion reactors. Therefore it is important to fully understand the release behavior of the tritium bred inside this material. This is not an easy task because of the complexity of processes occurring inside the grains, on the grain surfaces and inside the pores [1]. Among these processes, different surface reactions (such as adsorption, desorption and dissolution) need to be accurately described in order to determine the tritium release and inventory in Li₂O.

One of the problems that faces modeling is that no experimental results are present to determine the order of surface adsorption and desorption processes in solid breeder materials, with the exception of LiAlO₂ [2]. This led some models to consider them to be second order [3], while others use empirical equations to overcome this problem [4]. Although previous theoretical work was done to analyze the surface reactions in Li₂O [5], this work assumed the reactions were second order and did not investigate first order reactions. To date, no work was done to analyze first order reactions.

In this paper the order of the surface reactions is investigated to examine how it affects the steady state tritium inventories in the grain and on the surface at different conditions, thus deducing the correct order of surface adsorption and desorption.

2. The tritium inventory

The tritium inside Li-based breeders is divided mainly into two components: the surface inventory and the grain inventory, which is usually due to the tritium diffusion inside the grain. In Li₂O, an additional factor contributes to the grain inventory. This is the trapping of tritium atoms in the form of LiOT inside the grains.

2.1. The surface inventory

The surface inventory is controlled by four processes occurring on the surface. These are: 1. Adsorption of the atoms from the pore to the surface of the grains. For first order adsorption, the flux, R_{ads} is equal to [6]:

$$R_{ads} = k_{ads} \frac{C_p}{\sqrt{M}} (1 - \theta), \quad (1)$$

$$k_{ads} = \frac{\sigma \cdot z}{\sqrt{8 \times 10^{-3} \pi}} \sqrt{RT} \exp(-E_{ads} / RT), \quad (2)$$

where C_p is the concentration of the adsorbed species in the pore, θ is the total surface coverage (i.e. the fraction of filled surface sites). The term $(1 - \theta)$ represents the number of empty sites available for adsorption. E_{ads} is the activation energy of adsorption, R is the universal gas constant, T is the temperature, σ is the sticking coefficient, z is the number of sites adjacent to each atom, and M is the molecular weight of the adsorbing gaseous species. For second order adsorption [7]

$$R_{ads} = k_{ads,2} \frac{C_p}{\sqrt{M}} (1-\theta)^2, \quad (3)$$

$$k_{ads,2} = \frac{\sigma \cdot z}{\sqrt{8 \times 10^{-3} \pi}} \sqrt{RT} \exp(-2E_{ads} / RT). \quad (4)$$

Note that the squaring of $(1-\theta)$ and the doubling of E_{ads} stems from the fact that two atoms are required to adsorb in order to form H_2 , HT , or T_2 [7]

2. Desorption of atoms from the surface to the pore. For first order desorption, the desorption flux, R_{des} , is equal to [6]:

$$R_{des} = k_{des} \theta_i, \quad (5)$$

$$k_{des} = \frac{RN_s z T}{A_v h} \exp(-E_{des} / RT), \quad (6)$$

where E_{des} is the activation energy of desorption, θ_i is the surface coverage of the desorbing species, N_s is the number of sites on the surface of the grain, A_v is Avogadro's number, and h is Planck's constant. For second order desorption [7]:

$$R_{des} = k_{des,2} \theta_i^2, \quad (7)$$

$$k_{des,2} = \frac{RN_s z T}{A_v h} \exp(-2E_{des} / RT). \quad (8)$$

3. Dissolution of atoms from the surface to the grain. This flux, R_{diss} , is equal to [8]:

$$R_{diss} = k_{diss} \theta_i, \quad (9)$$

$$k_{diss} = \frac{RN_s z T}{A_v h} \exp(-E_{diss} / RT). \quad (10)$$

E_{diss} is the activation energy for dissolution.

4. A flux of atoms, R_β , going from the grains to the surface [6]:

$$R_\beta = k_\beta C_b (1-\theta), \quad (11)$$

$$k_\beta = \frac{1 \times 10^{13}}{\sqrt{N_s}} \exp(-E_\beta / RT), \quad (12)$$

where C_b is the tritium concentration in the grains just below the surface and E_β is the activation energy for adsorption from the bulk to the surface.

The four energies, E_{ads} , E_{des} , E_{diss} , and E_β are related through the activation energy of solution, E_s , and the heat of adsorption, Q [5].

$$E_s = E_{ads} - E_{des} + E_{diss} - E_\beta. \quad (13)$$

$$Q = E_{des} - E_{ads}. \quad (14)$$

The tritium surface inventory, I_s , is related to the tritium surface coverage, θ_T , by:

$$I_s = \theta_T N_s S_{BET} \rho V_{br} (1-\varepsilon), \quad (15)$$

where V_{br} and ε are the breeder volume and porosity, respectively, ρ is the theoretical density of the Li₂O and S_{BET} is the total BET surface area.

2.2. The grain inventory

The tritium inventory is divided into two parts: the inventory due to tritium diffusion and that due to trapping as LiOT

The tritium inventory, I_D , due to diffusion is equal to [9]:

$$I_D = \left[C_b + \frac{gr_s^2}{15D} \right] V_{br} (1-\varepsilon), \quad (16)$$

the tritium inventory, I_{tr} , due to trapping is equal to [9]:

$$I_{tr} = \left[C_b + \frac{gr_g^2}{15D} \right] \frac{k_{for}}{k_{dsn}} V_{br} (1 - \varepsilon), \quad (17)$$

where k_{for} and k_{dsn} are the reaction rate constants for formation and dissociation of LiOT [8]. Eqs. (16 and 17) can be combined together to give:

$$I_g = \left[C_b + \frac{gr_g^2}{15D} \right] V_{br} (1 - \varepsilon) (1 + K_{eq}), \quad (18)$$

where I_g is the tritium inventory inside the grain due to both diffusion and LiOT formation. K_{eq} is the equilibrium constant for the reaction $2LiOH = Li_2O + H_2O$ [8].

3. Surface coverage and grain concentration

As seen from eqs. (15 and 18), both the grain and surface inventories depend on the tritium surface coverage and tritium concentration in the grain. These in turn depend on the surface reactions. As will be shown, whether adsorption and desorption are first or second order reactions will greatly influence the tritium behavior and inventory on the surface and inside the grains.

Some observations were made when performing the following analysis. The first is that when protium is added to the purge gas, most of it is found in the form of H₂, whereas most of the tritium is found in the form of HT [10]. This is due to the fact that the amounts of tritium are much less than those of protium. This is also the reason why the surface coverage of the protium is usually much larger than that of the tritium [5]. These observations can be used to make the following assumptions:

1. $C_{pH_2} \gg C_{pHT}$
2. $C_{pHT} \gg C_{pT_2}$
3. $\theta_H \gg \theta_T$, or $\theta_{tot} \approx \theta_H$

where θ_{tot} , θ_H and θ_T , are the total, protium and tritium surface coverage, respectively. The above assumptions are valid for all the experiments.

3.1. First order adsorption/desorption

At steady state the desorbing and adsorbing fluxes of protium are equal, since it is not generated in the breeder. Therefore:

$$k_{des} \theta_H = k_{ads} \frac{C_{pH}}{\sqrt{M_{H_2}}} (1 - \theta_{tot}). \quad (19)$$

Since $\theta_{tot} \approx \theta_H$, we can simplify the above equation to give:

$$\theta_H = k_{ads} \frac{C_{pH}}{\sqrt{M_{H_2}}} \left[k_{des} + k_{ads} \frac{C_{pH}}{\sqrt{M_{H_2}}} \right]^{-1}. \quad (20)$$

For steady state tritium generation and release, the net flux coming out of the pores is equal to, g , the tritium generated inside the grains. Therefore

$$R_{des,T} - R_{ads,T} = g. \quad (21)$$

$$k_{des} \theta_T - k_{ads} \frac{C_{pT}}{\sqrt{M_{HT}}} (1 - \theta_{tot}) = g. \quad (22)$$

Solving eq. (22) for θ_T :

$$\theta_T = \frac{1}{k_{des}} \left[g + k_{ads} \frac{C_{pT}}{\sqrt{M_{HT}}} (1 - \theta_{tot}) \right]. \quad (23)$$

The tritium generation is also equal to the amount of tritium leaving the grains, i.e.

$$k_{\beta} C_b (1 - \theta_{tot}) - k_{diss} \theta_T = g, \quad (24)$$

or,

$$C_b = \frac{g + k_{diss} \theta_{tot}}{k_{\beta} (1 - \theta_T)}. \quad (25)$$

3.2. Second order adsorption/desorption

For second order surface processes, the same basic equations apply. However, the terms θ and $(1 - \theta)$ are replaced with θ^2 and $(1 - \theta)^2$. Equating different adsorbing and

desorbing fluxes containing protium, we get [5]:

$$k_{des,2}\theta_H^2 = k_{ads,2} \frac{C_{pH}}{\sqrt{M_{H_2}}} (1-\theta_{tot})^2. \quad (26)$$

Solving for θ_H , we get:

$$\theta_H = \frac{-k_{ads,2} \frac{C_{pT}}{\sqrt{M_{HT}}} \pm \sqrt{\left(k_{ads,2} \frac{C_{pH}}{\sqrt{M_{H_2}}}\right)^2 - \left(k_{ads,2} \frac{C_{pH}}{\sqrt{M_{H_2}}}\right) \left(k_{des,2} - k_{ads,2} \frac{C_{pH}}{\sqrt{M_{H_2}}}\right)}}{k_{des,2} - k_{ads,2} \frac{C_{pH}}{\sqrt{M_{H_2}}}} \quad (27)$$

Similarly for the tritium:

$$k_{des,2}\theta_H\theta_T - k_{ads,2}(1-\theta_{tot})^2 = g, \quad (28)$$

$$\theta_T = \frac{g + k_{ads,2} \frac{C_{pT}}{\sqrt{M_{HT}}} (1-\theta_H)^2}{k_{des,2}\theta_H}. \quad (29)$$

Eqs. (27 and 29) differ from those derived by Badawi et al. [5] in that they can be used for large values of θ_H whereas the equations in ref. [5] use the assumption that $\theta_H \ll 1$, and thus cannot be used for protium surface coverage more than 0.1.

The tritium grain concentration can be obtained from eq. (25).

4. Results and discussion

The tritium release from Li₂O was studied in a number of experiments. These include CRITIC-I [11-13] and BEATRIX-II [14-16]. The effects of purge gas temperature and composition on the tritium inventory were studied in those experiments. A summary of the sample and purge gas characteristics in CRITIC-I and BEATRIX-II experiments is shown in table 1.

The equations derived above were used to calculate the tritium inventory in Li₂O at different conditions, in order to reproduce the experimental results and to simulate the tritium behavior observed in those

Table 1
Summary of the experimental data used in the analysis.

Experiment	CRITIC-I [11-13]	BEATRIX-II Phase I [14-16]
Sample mass (g)	5.5 -6.9	11.95
Porosity (%)	8.5	20.7
BET area (m ² /kg)	52	60
Sample volume (m ³)	3.57 x 10 ⁻⁶	7.72 x 10 ⁻⁶
Grain radius (μm)	55	2.75
Tritium generation	5 Ci/day	5 Ci/day
Helium pressure (atm)	1	2.5
He flow rate (ml/hr)	100	100

experiments. The tritium inventory is found to exhibit the following behavior [11-16]:

1. it decreases with increasing temperature.
2. it decreases with increasing the hydrogen concentration in the purge gas.
3. it is controlled by the surface processes when the sample has small grains. For large-grained samples the diffusion is the rate-controlling mechanism.

4.1. Comparison with experimental results

As mentioned earlier, the tritium inventories using first and second order equations were compared with the experimental results. It should be noted, however, that two main factors will prevent the tritium inventory being accurately estimated using the derived equations. First, the tritium surface coverage is a function of the tritium concentration in the pore (see eqs. (23 and 29)). This changes along the pore, and thus changes the tritium surface coverage and the grain concentration along the breeder material. In the analysis, C_p was assumed to be constant and of known value. Second, the temperature was assumed to be constant in the analysis. However, in practice it changes along the samples.

Accurate calculation of the tritium inventory requires a numerical code in which the temperature is allowed to change along the solid breeder and the tritium pore concentration is calculated using the diffusion equation along the pore. However, the equations derived above can give a rough estimate of the tritium inventories.

In BEATRIX-II Phase I experiment, the changes in the tritium inventory, ΔI , corresponding to changes in the temperature and/or hydrogen concentration in the purge gas were measured. Nine cases were used for comparison, three with temperature changes and six with changes in H₂ concentration. Table 2 shows the results of the experiment and calculations using first and second order reactions.

Some observations can be made from Table 2:

1. There is a huge difference between ΔI calculated using first order reactions and those using second order reactions.
2. In cases with a changing purge gas composition (presented as H₂ pressure), the first order reactions do not predict any change in the tritium inventory.

3. The results using second order reactions are much closer to the experimental results than those using first order results.

In the CRITIC-I experiment, the change in tritium inventory was also measured when the temperature and/or hydrogen concentration in the purge gas were changed. Four cases were chosen for comparisons; two cases with purge gas changes and two with temperature changes. Table 3 shows the results of the experiment and calculations using both first and second order reactions. In all the cases, the changes in the tritium inventory calculated using first order reactions were much smaller than the experimental results. In the case when the hydrogen concentration in the purge gas changed from 0.01% to 0.1% there was no change in the calculated tritium inventory. The changes in tritium inventory using second order reactions were closer to those measured experimentally.

Table 2

Comparison between the changes in tritium inventory in CRITIC-I experiment and the calculations using first and second order reactions.

Experimental conditions	ΔI (Ci)	ΔI (Ci)	ΔI (Ci)
	Experiment	Second order	First order
550°C, pure He → He + 0.01% H ₂	13	11.4	8.3 x 10 ⁻⁵
550°C, He + 0.01% H ₂ → He + 0.1% H ₂	5	0.4	0
He + 1% H ₂ , 651°C → 638°C	+ < 0.1	0.5	2.8 x 10 ⁻⁴
He + 1% H ₂ , 638°C → 730°C	- < 0.1	-0.2	-2 x 10 ⁻⁵

Table 3

Comparison between the changes in tritium inventory in BEATRIX-II experiment and the calculations using first and second order reactions.

Temperature	H ₂ pressure (Pa)	ΔI (mCi)	ΔI (mCi)	ΔI (mCi)
		Experiment	Second order	First order
638°C → 600°C	150	28 ± 54	96	0.2
600°C → 550°C	150	36 ± 27	44	1
550°C → 597°C	150	-24 ± 19	-34	-0.3
636°C	150 → 0	15 ± 10 x 10 ³	300	0
636°C	0 → 15	-3 ± 10 x 10 ³	-124	0
636°C	15 → 150	-5 ± 10 x 10 ³	-176	0
545°C	150 → 0	15 ± 10 x 10 ³	717	0
545°C	0 → 15	-4 ± 10 x 10 ³	-388	0
545°C	15 → 150	-7 ± 10 x 10 ³	-329	0

4.2. Effect of temperature

The tritium inventory was calculated for temperatures in the range 500K – 1200K using data from both experiments. The results are shown in figs. 1 and 2 for CRITIC-I and BEATRIX-II data, respectively. Both first and second order processes show that the tritium inventory decreases with increasing temperature. However, the tritium inventory for second order processes is much higher

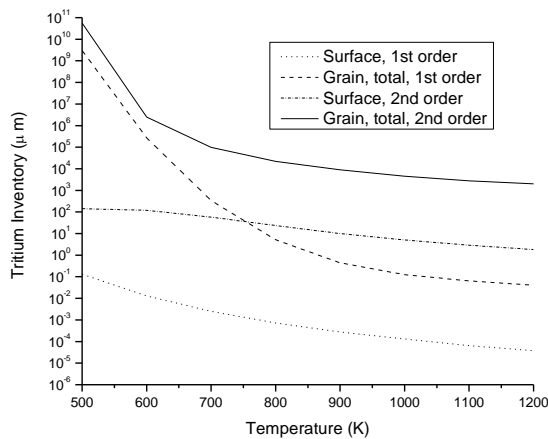


Fig. 1. Effect of temperature on the tritium inventory using first and second order reactions and CRITIC-I data with He + 0.01%H₂.

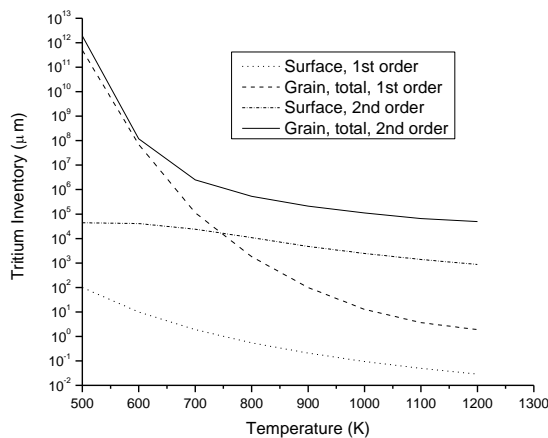


Fig. 2. Effect of temperature on the tritium inventory using first and second order reactions and BEATRIX-II data with He + 0.01%H₂.

than that for first order. In fig. 1 the second order inventory is found to be one order of magnitude higher at 500K and four orders of magnitude higher at 1200K. Note that both the second and first order inventories are too high at 500K (5.6×10^4 Ci and 3×10^3 Ci, respectively). This temperature is too low for use in the fusion reactor because the grain diffusion becomes too slow and the LiOT formation causes a large fraction of the generated tritium to be trapped inside the grains.

Fig. 2 shows that the tritium inventory using the BEATRIX-II data is higher than in fig. 1 (with the CRITIC-I data). This is due to the higher tritium generation in BEATRIX-II. The second order inventory is also higher than the first order inventory by one order of magnitude at 500K and by four orders of magnitude at 1200K. The reason why the difference in tritium inventory is smaller at lower temperatures is because at low temperatures the tritium release is diffusion controlled, whereas at higher temperatures it is surface controlled. Thus whether the surface reactions are first or second order will have a greater impact at higher temperatures.

4.3. Effect of Purge gas composition

The tritium inventory was calculated for different protium concentrations inside the purge gas, ranging from 0.0001% to 50%. The tritium inventory calculated using data from CRITIC-I and BEATRIX-II experiments are shown in figs. 3 and 4, respectively. Figure 3 shows that the first order inventory is independent of the protium concentration. The tritium inventory is 5.2 μCi in all cases. When second order surface reactions are used, the tritium inventory is found to decrease with increasing the H₂ concentration. At low H₂ concentration, the change in the tritium inventory is more than that at high H₂ concentration. Changing the percentage of H₂ in the purge gas from 0.0001% to 0.001% changes the inventory by one order of magnitude (0.22 Ci to 0.07 Ci, respectively). Changing it from 1% to 10% changes the inventory by 30% (2210 μCi to 712 μCi, respectively).

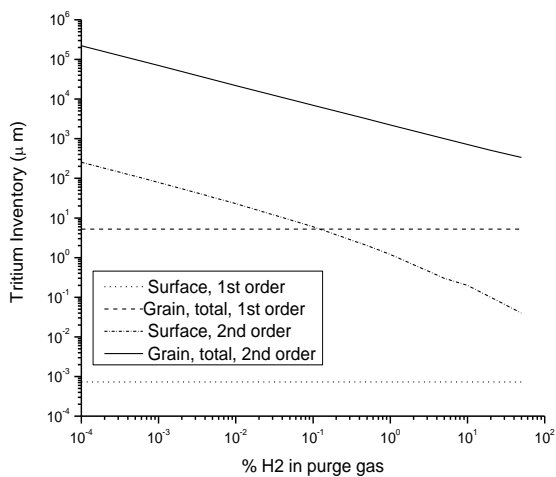


Fig. 3. Effect of H₂ concentration on the tritium inventory using first and second order reactions and CRITIC-I data with 800K.

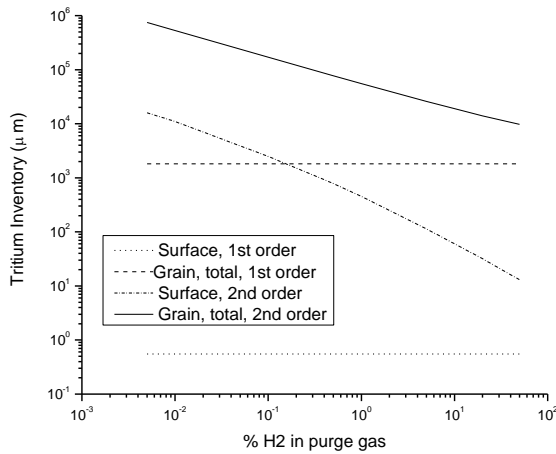


Fig. 4. Effect of H₂ concentration on the tritium inventory using first and second order reactions and BEATRIX-II data with 800K.

The tritium inventory using data from BEATRIX-II experiment is shown in fig. 4. Again, the first order inventory is independent of the protium concentration. It is 1.8mCi in all cases. The second order inventory is shown to decrease with increasing the H₂ concentration.

4.4. Effect of grain size

The tritium inventory was calculated using data from both experiments, with Li₂O grain

sizes changing between 0.01μm and 5 mm. The results are shown in figs. 5 and 6 for CRITIC-I and BEATRIX-II data, respectively, using first and second order reactions.

As seen in fig. 5, the inventory is found to increase with increasing grain size. Both the first and second order surface inventories are found to be independent of the grain radius. For second order reactions, the grain inventory is 40 μCi for 0.01 μm grains. The surface inventory is 23μCi. Using first order reactions with the same grain size gives a grain and surface inventories of 5.4 x 10⁻⁴μCi and 7.3 x 10⁻⁴ μCi. The grain inventory increases with grain size.

For 0.1μm grains, the second order grain and surface inventories are 40 μCi and 23 μCi. The first order grain and surface inventories are 5.4 x 10⁻⁴μCi and 7.3 x 10⁻⁴ μCi. The inventories of the small grains suggest that the tritium release is surface controlled. This is due to the fact that the small radius allows the tritium to diffuse fast from the grains. Larger grains makes the tritium take a longer time to diffuse to the surface, thus making the release diffusion control.

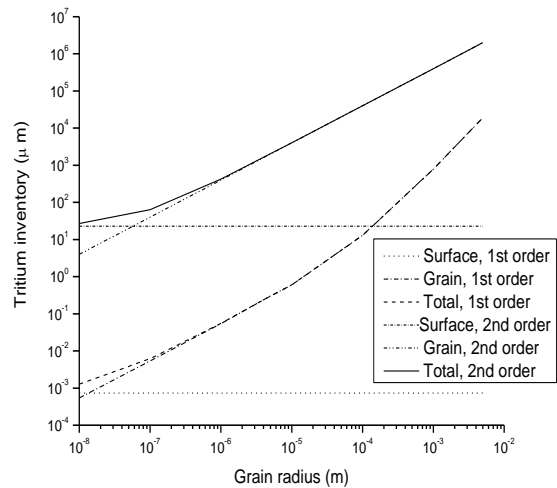


Fig. 5. Effect of grain radius on the tritium inventory using first and second order reactions and CRITIC-I data with 800K and He + 0.01% H₂.

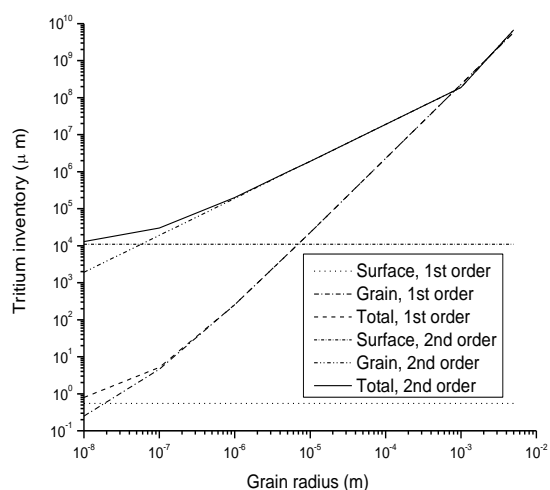


Fig. 6. Effect of grain size on the inventory using second order reactions and BEATRIX-II data with 800K, He + 0.01%H₂.

Fig. 6 shows the results using BEATRIX-II data. Again, the surface inventories are independent of the grain size, whereas the grain inventories increase with increasing the grain size. The first order inventories are also much smaller than the second order inventories. The difference between the two inventories are much larger for smaller grains. The first order inventory is about five orders of magnitude smaller than the second order inventory for 0.01 μm grains (0.8 μCi and 13 mCi, respectively). For 5000 μm grains, the first order inventory is about 15% smaller than the second order inventory (5800 Ci and 6800 Ci, respectively). The huge difference at small sizes can be attributed to the fact that the surface reactions greatly influence the tritium release for small-sized grains whereas the grain diffusion controls the tritium release for large-sized grains.

5. Conclusions

The first and second order reactions were used to reproduce the experimental results and to simulate the tritium behavior observed in CRITIC-I and BEATRIX-II experiments. The experimental data included cases with changing temperature and changing H₂

concentration. From the analysis, the following conclusions can be made:

1. The first order reactions give a tritium inventory that is much smaller than that measured experimentally. The second order reactions give results that are closer to the experiments, the worst case being off by one order of magnitude.
 2. Both first and second order reactions give the same response to temperature changes. In both cases the tritium inventory decreases with increasing temperature. The difference is in the numerical values of the inventory, which are much smaller in the case of first order reactions.
 3. The first order reactions do not simulate the behavior related to changes in purge gas composition, since they give inventories that are independent of the H₂ concentration. Only second order adsorption/desorption reactions are able to simulate experimentally observed trends.
 4. At very low temperatures, the first and second order inventories are comparable, showing that the tritium release is diffusion-controlled. At high temperatures the inventories differ by 4 orders of magnitude, which means that they are surface-controlled.
 5. Both first and second order reactions give inventories that increase with increasing grain size. The second order inventories are surface controlled at larger grains than the first order inventories (about a factor of 5).
- This leads to the conclusion that the adsorption and desorption of tritium on the surface of Li₂O are second order reactions.

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