RECENT ADVANCES IN THE DEVELOPMENT OF SOLID BREEDER-BLANKET MATERIALS

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Increasing attention in breeder blanket development has been given to the lithium-containing ceramic materials. The most promising of these materials include Li_2O , Li_8ZrO_6 , Li_4SiO_4 , and $\gamma\text{-LiAlO}_2$. Recent studies have focused on Li_2O because of its high tritium breeding potential and good thermal characteristics. Tritium solubility in Li_2O is within acceptable ranges and this oxide displays excellent behavior under neutron irradiation. A broad scope of laboratory and in-reactor irradiation experiments are underway to further investigate these materials.

1. INTRODUCTION

A current design¹ for fusion reactors includes independent production of tritium by introducing a tritium breeding blanket as part of the first wall design. The breeding blanket is one of the most important components of a fusion reactor because it directly involves both energy extraction and tritium breeding, both of which are critical to development of fusion power. Because of their overall desirable properties, lithium-containing ceramic-type solids (e.g., Li_2O , $\gamma\text{-LiAlO}_2$, Li_4SiO_4 , and Li_8ZrO_6) are receiving increasing attention as possible breeder materials. A recent workshop² focused attention on the limited data base of these materials and stimulated the initiation of laboratory and in-reactor irradiation studies to improve our general understanding of them. Solid breeder materials also received considerable attention at an IAEA workshop.³ A just-published interim report⁴ on a blanket comparison study also affirms the potential for the lithium-containing ceramic materials to serve as tritium breeders for fusion reactors.

Recent research emphasis has given greatest attention to Li_2O because of its high tritium breeding potential. Therefore, much of this paper focuses on that material. Continuing research efforts will expand the data base for all candidate materials.

2. BLANKET DESIGN

Temperature Limits

The tritium inventory in a candidate breeder material, which is characterized by transport (diffusion) and solubility (thermodynamic), is predicted to

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possess a strong temperature dependence and to be sensitive to diffusion path length. In most reactor designs using solid breeder materials, the blanket is fixed and tritium recovery is achieved by sweeping a purge gas through porosity in a bed of the solid. The operating temperature of the breeder blanket must be high enough that acceptable tritium transport characteristics are achieved yet low enough that sintering, vaporization, etc., do not lessen blanket performance. We are recommending operating temperature limits based upon an assessment of materials properties that may affect on either the upper or lower temperature bound ($T_{\max}$ or $T_{\min}$).

Bulk diffusion is considered to be a significant contributor to the tritium inventory in a blanket material. As tritium diffusion slows, both residence time and inventory of the tritium will increase. While the selection of an inventory limit is somewhat arbitrary, candidate materials exhibiting low inventory are clearly more desirable. Available data for the candidate materials suggest $T_{\min}$ between 300 and 410°C (see Table I). Below these temperatures, blanket inventory is likely to increase dramatically.

Except for Li_2O , $T_{\max}$ for solid breeders (700-1400°C, Table I) is based on experimental determination of the temperature at which closed porosity is apparent, this condition occur at approximately 90% of the theoretical density. In general, the thermal sintering characteristics of stable ceramic oxides are quite similar. Temperatures in excess of $0.8 T_m$ (T_m = the absolute melting point) are required before significant thermal sintering occurs. However, for some ceramic oxides, neutron irradiation is known to decrease the temperature at which sintering occurs. The effects of radiation may, therefore, close porosity at temperatures lower than $0.8 T_m$, suggesting that lower values of $T_{\max}$ would be needed.

There are important conclusions to be drawn from the thermomechanical studies on Li_2O . It is evident that the solubility of LiOH in Li_2O is very low and the phases are nearly immiscible. Consideration of this factor is important because the blanket must not operate in a regime where LiOH is the stable phase. At high (>800°C) temperatures, LiOT vaporization could present some design challenges in managing potential Li_2O transport. However, high temperatures are very attractive from a thermal-hydraulic design point of view. Thus, designs which take advantage of a high $T_{\max}$ must have purge gas flow directed only to the "cold" region (<700°C) of the breeder.

3. MATERIALS PROPERTIES

3.1 Tritium Solubility

Single Phase System

Experimental measurements on the solubility of water vapor with solid Li_2O at temperatures of 973 to 1273 K have been completed.⁵ Least squares fitting of the resulting data yielded the following analytical expression for the concentration of dissolved LiOH (expressed as mol fraction, x) as a function of temperature (in K) and partial pressure of water vapor (in atm):

$$\log(X_{\text{LiOH}}) = (0.427 + 1.700 \times 10^{-4} T) \log(p_{\text{H}_2\text{O}}) + (-17.667 + 2.502 \times 10^{-2} T - 9.62 \times 10^{-6} T^2) \quad (1)$$

Assuming there are no isotope effects, this expression may be used to compute the solubility of LiOH in Li₂O. Figure 1 is a plot of the relationship between concentration of LiOH (X=H or T) as a solute in solid solution in Li₂O and the partial pressure of H₂O. These curves indicate that the solubility of LiOH increases with temperature at constant partial pressure of water. Calculations using an ideal solution model with JANAF thermochemical data indicated that the solubility should decline with temperature, contrary to observations. This discrepancy has been resolved with the realization that the LiOH-Li₂O system is thermodynamically non-ideal. Calculations utilizing activity coefficients derived from the solubility data yield solubilities that are in agreement with the observed behavior. Generally similar results were obtained in the experimental study of Norman and Hightower⁶. At this time, comparison between the two studies^{5,6} is easiest in terms of activity coefficients; further discussion of the activity coefficients appears below.

Two Phase Systems

Experimental work⁷ has been completed to determine the partial pressure of water vapor required to precipitate LiOH from Li₂O in the temperature region (573 to 894 K) above and below the melting point of LiOH (744 K). The current data are in good agreement with earlier work⁸ on this system below the melting point of LiOH (723 K). Two equations represent the Tetenbaum and Johnson data. Below the melting point of LiOH, the minimum water vapor pressure required to form a two phase system is given by:

$$\log p(\text{H}_2\text{O}, \text{g, atm}) = -6718/T + 6.45 \quad (2)$$

For example, at 700 K, $p(\text{H}_2\text{O}) = 7.1 \times 10^{-4}$ atm. Above the melting point of LiOH, the critical water vapor pressure is given by:

$$\log p(\text{H}_2\text{O}, \text{g, atm}) = -4406/T + 3.27 \quad (3)$$

At 800 K, $p(\text{H}_2\text{O}) = 5.8 \times 10^{-3}$ atm and at 900 K, $p(\text{H}_2\text{O}) = 2.4 \times 10^{-2}$ atm. Thus, the maximum allowable partial pressure of H₂O to avoid second phase formation, is, at 700 K, i.e., 710 vppm. Less stringent conditions prevail at higher temperature.

There is a gap of about 80 degrees between the upper end of the temperature range of these data and the lower end of the temperature range for the single phase solubility data mentioned earlier. It is probably more reliable to extrapolate the two phase data to higher temperatures to obtain an estimate of the water vapor pressure required to saturate the Li₂O with LiOH than to extrapolate the single phase solubility and activity coefficient data to lower temperature.

Activity Coefficients

Activity coefficients for LiOH (γ) as a solute in Li₂O were derived from a combination of the Tetenbaum et al.⁹ data on the concentration of LiOH as a

TRITIUM SOLUBILITY IN Li_2O AS LiOT ; 1 ATM. TOTAL PRESSURE

function of composition and temperature, and thermochemical data for LiOH(l) , LiOH(g) , $\text{H}_2\text{O(g)}$, and $\text{Li}_2\text{O(s)}$ from the JANAF tables. The resulting expression is:

$$\log(\gamma) = (21.517 - 3.0263 \times 10^{-2} T + 1.1392 \times 10^{-5} T^2) + (3.1703 \times 10^{-2} - 7.3830 \times 10^{-5} T) \log(p\text{H}_2\text{O}) \quad (4)$$

The range of partial pressures in the experimental work was 10^{-5} to 10^{-3} atm. The rather large activity coefficient values at lower temperatures ($\sim 10^3$) are noteworthy. Over the concentration regime studied, the activity coefficients are greater than unity, indicative of positive deviation from ideality. Such deviations from ideality are interpreted to result from repulsive interactions between LiOH and Li_2O , which is suggestive of low solubility of LiOH in Li_2O (i.e., lower than would be predicted by an ideal solution calculation).

The Norman and Hightower study⁶ has yielded the following activity coefficient expression, using the Gregory and Mohr⁸ data as reference values for the activity of pure LiOH :

$$\log(\gamma) = 4867/T - 0.808 + 1.27 \times 10^{-3} T - 1.051 \log T \quad (5)$$

This equation resulted from application of a regular solution theory expression to the data and is distinguished from Eq. 4 by being independent of the partial pressure of water vapor. However, at temperatures above 700°C the two sets^{6,9} of data overlap for experimental errors considered at the two sigma level.

3.2 Mechanical

Experimental data on the mechanical properties of solid breeder materials are essentially nonexistent at the present time. Baseline information is essential to the design of solid breeder blankets and is necessary to understand the irradiation behavior of solid breeders. Elastic properties are needed to determine the thermoelastic mechanical behavior of materials, i.e., Young's modulus (E) and Poisson's ratio (ν). Thermal stresses in the solid breeder are directly proportional to E and are affected by ν , depending on the solid breeder configuration, i.e., whether the configuration approaches a plane-strain or plane-stress condition. Since thermal stresses typically vary from tensile to compressive in the solid breeder under a temperature gradient or during temperature cycling, the possibility of tensile-stress-induced cracking must be considered. The cracks can represent sources for localized stress and strain in the cladding if cladding is used to contain the solid breeder. In the event of solid breeder swelling and/or ratchetting during blanket operation, the localized stress and strain is a potential cause of failure for the cladding materials. Similarly, ceramic brittle materials are often observed to fracture at a critical uniaxial tensile stress. This maximum stress (σ_f) can be used as a fracture criterion to determine whether or not cracking will occur.

3.3 Tritium Transport

The rate and method of tritium transport in a ceramic breeder material are important considerations for determining tritium inventory. Unfortunately, the literature¹⁰⁻²⁰ reporting diffusion coefficient data have, in most cases, neither characterized the grain size nor identified the migrating species type. The modeling of tritium transport and release from solid breeder materials must recognize the relative importance of mass transport in the gas phase, as well as solid state diffusion. Experimental investigations on oxygen containing compounds have produced considerable uncertainty in the absolute value of the tritium diffusion coefficient and minimal data in characterizing the transfer of tritium from the gas-solid interface into the gas stream. The most extensive and well-characterized work has been performed on Li_2O . Figure 2 shows the temperature dependence of the diffusion coefficient for tritium in Li_2O , as well as for other relevant species, i.e., oxygen, lithium, hydrogen, and deuterium. A model for tritium diffusion in Li_2O has been suggested consistent with existing diffusion data.²¹ Careful analysis of the data suggests that there exists a systematic pattern that can be interpreted in terms of the following diffusion mechanism.

Neutron irradiation converts the ^6Li components in Li_2O into tritium and helium, creating at the same time (among other defects) a Li vacancy (V_{Li}). The origin of the V_{Li} can arise from several sources: (1) the extrinsic impurity-induced defects that control Li diffusion below 1000°C , (2) the $^6\text{Li}(n,\alpha)\text{T}$ reaction that generates many defects in transforming Li into ^3H and ^4He atoms, and (3) defects generated by displacement damage, i.e., recoil of the energetic ^3H and ^4He atoms.

The following defect reaction takes place subsequently:

The notation V_{Li}/T denotes a stable defect complex comprising a V_{Li} and a tritium atom bound together in a certain geometrical configuration. This concept could be useful when discussing the chemistry of tritium release from Li_2O . However, the migrating component of this unit is the complex V_{Li}/T . In this defect complex, one expects $E_{V_{\text{Li}}/\text{T}} \approx E_{V_{\text{Li}}}$ since interstitial tritium should have a lower migration energy than V_{Li} . This interpretation is consistent with the tritium diffusion data. Below 400°C the slope of $\ln D_{\text{T}}$ vs. $1/T$ is almost the same as that of the slope of $\ln D_{\text{Li}}$ vs. $1/T$.

On the other hand, this V_{Li}/T complex has only a finite free energy of binding (ΔG_b). If the temperature is raised above 400°C such that $|\Delta G_b| \lesssim k_{\text{B}}T$, then substantial dissociation into V_{Li} and tritium takes place. Thereafter, the tritium migrates mainly in a form that is not associated with Li vacancies, which should have a lower migration energy (i.e., lower slope) than the low-temperature regime. This hypothesis is again consistent with the data. The temperature above which substantial break-up of the V_{Li}/T complex takes place appears to be 500°C . According to this model, there should be a temperature regime (between 400 and 500°C) where both V_{Li}/T and interstitial tritium make a substantial contribution to tritium release from Li_2O . There should then be a non-Arrhenius behavior in that regime. This prediction agrees very well with the qualitative trend indicated by the Nasu data set, which is the only one that spans the critical temperature regime.

TEMPERATURE DEPENDENCE OF DIFFUSION
COEFFICIENTS OF TRITIUM, DEUTERIUM, HYDROGEN,
LITHIUM, AND OXYGEN IN LITHIUM OXIDE

3.4 Blanket Fabrication

There are two possible blanket configurations for solid breeders: (1) pressed and sintered pellets and (2) sphere-pac (spherical particles). The pressed and sintered technique has been used successfully for preparing Li_2O , Li_4SiO_4 , $\gamma\text{-LiAlO}_2$, and Li_2ZrO_3 . Excellent experience was gained recently in preparing large quantities of sintered Li_2O pellets for the TFTR lithium blanket module (LBM) program.²² Sphere-pac technology has been developed for LWR and FTR fuels and is considered attractive for solid breeders.

Cold pressing and sintering techniques were developed for Li_2O , Li_4SiO_4 , and $\gamma\text{-LiAlO}_2$ samples irradiated in ORR.²³ For the EBR-II/FUBR irradiation experiment, techniques were developed to control impurities, structures, and grain size for candidate material powders and to fabricate these powders into pellets by hot pressing techniques.²⁴ Li_2O pellets of 70 to 93% theoretical density were prepared using cold pressing and sintering techniques.²⁵ Dense pellets (up to 99% theoretical density) were prepared by the same authors using hot isostatic pressing methods. Current efforts suggest that grain size can be best preserved using hot pressing methods.

For fission fuels the gel-sphere-pac preparation²⁶ consists of three major steps: (1) preparation of a sol or special solution ("broth"), (2) gelation of droplets of sol or broth to give semi-rigid spheres, and (3) drying and sintering these spheres to a high density. The sphere-pac configuration requires three sizes of spheres to achieve about 83% smear density. These sizes have diametral ratios of about 40:10:1. The actual diameters currently favored are 1200, 300, and 30 μm .²⁷ The process for loading sphere-pac fuel into fuel rods involves vibratory compaction. Considerable technology has been developed regarding blending ratios and loading sequences to produce maximum smear densities and minimum loading times.

Little has yet been done with fabrication of materials of interest for breeder blankets. A method for producing porous lithium oxide spheres has been patented by Research Dynamics, Inc.²⁸ In this process, molten lithium hydroxide passes through a small bore tube to produce molten microspheres that are cooled in an inert atmosphere. The microspheres are then placed in a vacuum at 200°C for conversion to Li_2O . A similar method may be applicable for producing high-density solid breeder microspheres.

A major advantage of the sphere-pac configuration over pressed and sintered materials is the reduction in thermal contact resistance between the breeder material and its containment, thereby improving overall thermal conduction. This reduced thermal contact resistance can significantly change the temperature distribution of the breeder material. The quantification of this reduction in contact resistance and improvement in effective conductivity for a particular material needs to be confirmed.

4. IRRADIATION BEHAVIOR

Recent in-reactor tests²⁹⁻³² have shown that the candidate breeder materials perform well although their current irradiation times are measured in months.

rather than years. Available data indicate that tritium retention is low and pellet integrity is reasonably good. Vaporization and swelling introduce potential $T_{\max}$ and lifetime-limiting problems for Li_2O , whereas other materials do not appear as limited.

For current designs, Li_2O has a recommended $T_{\max}$ of 800°C or higher, depending upon the design approach selected. This upper limit is now established by vaporization mass transport as opposed to an earlier limit associated with tritium release mechanisms, i.e., sintering, grain growth, etc. Irradiation data show the occurrence of grain growth in Li_2O at temperatures above 700°C , but the data also imply that there the dependence of tritium release on grain growth is not as strong as earlier suggested.

The tritium inventory limit of a blanket (see Table II) is not represented here as a single quantitative value, for example 10 kg, since other factors such as lifetime and tritium production rates directly affect the inventory. Instead, the tritium inventory is considered on a percentage basis. From this viewpoint, a 2% inventory at the end-of-life represents a 2% burden on the breeding ratio requirements of a specific blanket design.

Hence, the tritium retention data for Li_2O indicate that under worst-case conditions (TULIP: large grains, moderate temperatures and static-plena) tritium inventories of the order of 9% were accommodated. Under more favorable conditions (FUBR-1A: finer grain size and higher temperatures), much lower tritium burden was found. The optimum condition occurred in the TRIO experiment wherein only 0.003% retention was found. Tritium recovery from the removed blanket material at end-of-life would further reduce the impact of retention.

The integrity of monolithic ceramics bodies featured in some design concepts take on importance when tight temperature limits are imposed for materials like Li_2O . Pellet cracking and material rearrangement have resulted in uncertainties in the temperature distribution within the blanket. During the irradiation experiments completed to date, pellet integrity appeared good for all the materials considered, but internal crack generation has been observed; however, large thermal gradients did not develop in the pellets tested. The cracks observed were found to be generally parallel to the direction of heat transfer, and thus, would have a minimal impact on operating temperature.

Vaporization and redeposition on cladding walls of approximately 3% of the Li_2O was observed in the FUBR-1A experiment at an estimated temperature of 900°C and a burnup of 3×10^{20} captures/ cm^3 . The transport was most probably in the form of LiOT vapor. Consequently, solid breeder designs with flow channels operating at temperatures above 800°C must accommodate to some solid breeder material redistribution. Purge gas containing the vaporized LiOT species can transport material to cooler regions downstream, where condensation/deposition of LiOT could result in flow blockage. It is not entirely clear what level of transport is permissible in this case. On the other hand, if designs locate flow channels in only the low temperature regions of the blanket ($T < 700^\circ\text{C}$), then LiOT transport distances are reduced and potential problems considerably reduced. In a design having low-temperature flow channels, transport would be radially along the temperature gradient within the solid breeder, and deposition would be on other Li_2O grains that were

operating at low temperatures ($T < 700^{\circ}\text{C}$). This deposition could restrict gas permeability somewhat, but is not likely to produce the blockages considered for the high temperature flow channels.

Swelling data on lithium ceramics in the FUBR-1A experiments indicate that swelling may be the most undesirable property of Li_2O in comparison to other ceramics. In that experiment, Li_2O pellets, did not densify through sintering but swelled in an inhomogeneous manner to approximately 2% diametrical. Since swelling is thought to be caused by defect and helium production, it must be assumed that swelling will be proportional to burnup and will increase at higher burnup levels.

Material swelling during irradiation has provided lifetime-limiting problems for some materials in fission reactor applications, i.e., graphite, stainless steel, etc. In other situations, however, swelling does not limit the lifetime of the material because it can be restrained by surrounding components, the most notable example of this situation is the restraint of mixed oxide fuel by stainless steel cladding. If Li_2O can plastically deform sufficiently at blanket operating temperatures, swelling could be restrained and perhaps not constitute a life-limiting problem. If it cannot be restrained, designs must either accommodate to this property or accept lower lifetimes. Other ceramics, such as LiAlO_2 , Li_4SiO_4 , and Li_2ZrO_3 , do not appear to swell and thus are not limited in the same way.

5. RESEARCH FOCUS

Because of their overall desirable properties, the lithium containing ceramics are receiving increasing attention as possible breeder material for fusion reactors. Recent research efforts have improved our understanding of these ceramics; however, because the data base for the lithium-containing ceramics is not large, the present research effort is concerned with the measurement and compilation of physical, mechanical, and thermodynamic properties of candidate solid breeder materials and with determination of the effects of neutron irradiation on them. Work is in progress on determining the tritium transport mechanism within candidate materials (including the diffusion coefficient for tritium in these solids), measuring the solubility of hydrogen (tritium), and characterizing the phase relationships in breeder-material/hydrogen systems.

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Table I. Property Values and Recommended Temperature Limits
for Candidate Solid Breeder Materials^a

	Properties			Recommended Temperature Limits, °C		
	MP, °C	ρ_{Li} , g/cm ³	K, ^b W/mk	T _{min}	T _{max}	ΔT
Li ₂ O	1433	0.93	3.4	410 ^d	800 ^{c,h}	390
γ -LiAlO ₂	1610	0.28	2.2	300 ^d	1200 ^f	900
Li ₅ AlO ₄	1047	0.61	2.3	350 ^d	780 ^g	430
Li ₂ SiO ₃	1200	0.36	1.5	410 ^d	1000 ^f	590
Li ₄ SiO ₄	1250	0.54	1.5	320 ^d	950 ^f	630
Li ₂ ZrO ₃	1616	0.33	1.3	400 ^e	1400 ^f	1000
Li ₈ ZrO ₆	1295	0.68	1.5	350 ^e	980 ^g	630
Li ₂ TiO ₃	1550	0.33	2.0	400 ^e	1185 ^g	785

^aSeparate effects data not available; therefore, effects of radiation on temperature limits are unknown and not reflected in this table.

^bEstimated for 85% smeared density and temperature of 1000 K.

^cEstablished from chemical considerations, i.e., reaction with moisture to form LiOH. $\log p(\text{LiOH}, g)(\text{atm}) = -8635/T + 1/2 \log p(\text{H}_2\text{O}, g) + 4.57$.

^dEstablished from diffusion/inventory considerations.

^eEstimated assuming similar properties.

^fEstablished experimentally from onset of closed porosity.

^gEstimated assuming $T_{\text{max}} = 0.8 T_m$, K.

^h1000°C for design approaches with helium purge gas flow directed only to the "cold" region of the breeder.

Table II. Irradiation Data on Candidate Ceramic Breeder Materials

Experiment	Material	Temperature	(10 ²⁰ Maximum Burnup captures/cm ²)	Tritium Retained	
				%	wppm
OG-5/ORR(29)	γ -LiAlO ₂	850°C	10.3	0.003	<0.01
	Li ₂ O	600°C	0.3	0.5	~1
	Li ₄ SiO ₄	600°C	0.3	0.5	~1
TULIP/EBR-II(30)	Li ₂ O	600°C	3	<8	110
	Li ₂ O	600°C	11	<4	165
FUBR/EBR-II(31)	Li ₂ O	500°C	3	<9	90
	Li ₂ O	700°C	3	<3	30
	Li ₂ O	900°C	3	<1	10
TRIO-1(32)	LiAlO ₂	650°C	2.2	0.003	<0.1

DISCLAIMER

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