# 11. Molten salt

## 11.1 Flibe Molten Salt Research for Tritium Breeder Applications

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This paper presents an overview of Flibe ($2LiF \bullet BeF_2$) molten salt research activities conducted at the INEEL as part of the Japan-US JUPITER-II joint research program. The research focuses on tritium/chemistry issues for self-cooled Flibe tritium breeder applications and includes the following activities: (1) Flibe preparation, purification, characterization and handling, (2) development and testing of REDOX strategies for containment material corrosion control, (3) tritium behavior and management in Flibe breeder systems, and (4) safety testing (e.g., mobilization of Flibe during accident scenarios). This paper describes the laboratory systems developed to support these research activities and summarizes key results of this work to date.

### 1. INTRODUCTION

The molten fluoride salt $2LiF \bullet BeF_2$, commonly referred to as Flibe, has been proposed for use in self-cooled tritium breeders for both magnetic and inertial confinement fusion applications [1-6]. Flibe has been considered for these applications because it has good heat transfer properties, has low electrical conductivity leading to small MHD effects, is relatively inert to air or water compared to liquid metals, and it has good neutron attenuation characteristics. However, it has several disadvantages that require attention and design mitigation, namely, the potential for high corrosion rates in containment materials, the potential for high tritium permeation losses through containment structures, and the chemical toxicity of beryllium. The corrosion potential for molten Flibe relates to the presence of impurities in the salt and to free (or excess) fluorine that is generated via nuclear transmutation reactions in the salt components [4-6]. As demonstrated during development and operation of the Molten Salt Reactor Experiment, (MSRE), corrosion control can be managed by suitable REDOX (reduction/oxidation) chemistry in the salt [7,8]. Based on prior measurements [9], the solubility of $H_2$ and $D_2$ is very low in pure Flibe, about a factor of 100 lower than that for HF and DF in Flibe at 600°C [10]. Therefore with good REDOX control in the Flibe, tritium will be released in elemental phase to high-temperature containment structures that can experience significant permeation unless mitigation measures are taken. Even though

Flibe is relatively inert, the high chemical toxicity of beryllium warrants an investigation into mobilization behavior of Flibe under accident conditions such as a breach of the containment structures that expose molten Flibe to air and moisture.

An international research effort has been established as part of the second Japan/US Program on Irradiation Tests for Fusion Research (JUPITER-II) with specific objectives to address the above tritium/chemistry and safety issues for Flibe use in tritium breeders [11,12]. The goals of this research are as follows: (1) development of the capabilities for preparing, purifying, characterizing, and handling 1-liter size quantities of Flibe for use in experiments, (2) development of a REDOX agent and demonstration of its ability to control corrosion in a Flibe blanket, (3) characterization of tritium behavior in REDOX-controlled Flibe and development of a tritium control strategy for a molten Flibe blanket in a fusion power plant, and (4) characterization of physico-chemical forms of material mobilized during an accidental spill of Flibe and development of Flibe safe handling practices. Participants in this research come from various Japanese universities, with much of the work done in the Safety and Tritium Applied Research (STAR) Facility [13] at the Idaho National Engineering and Environmental Laboratory (INEEL).

The purpose of this paper is to provide an overview of the research activities to date. Each of the goals stated above will be addressed by describing laboratory systems developed to support the research activities and by summarizing key results of the work to date.

## 2. FLIBE PREPARATION

We have established the capability at the INEEL to prepare, purify and characterize approximately 1-liter quantities of Flibe per batch. The salt is prepared from reagent-grade LiF and $BeF_2$. Typically, the component powders are dried in an inert environment at temperatures of about 250°C before they are weighed and mixed to provide the 2:1 mole ratio of Flibe. The powder mixture is then melted in an inert environment at temperatures of 600 to 800°C, allowing the liquid to thoroughly mix before cooling to ambient temperatures. This work is done in inert gas gloveboxes to ensure environmental integrity for the material handling and to prevent the spread of Be-bearing particulate in the worker environment.

Photos of some of the equipment used to prepare the Flibe are shown in Figures 1-2. Component materials are placed inside a crucible, either glassy carbon or nickel. The loaded crucible is placed inside a type-316 stainless steel pot that is equipped with penetrations and isolation valves to enable inert gas purge through the pot during thermal cycle treatments (see Figure 1). This work is done inside a glovebox that is purged with dry, ultra-high-purity helium. The loaded pot is transferred to a second glovebox that is equipped with pot heater assemblies capable of heating the pots to 800°C and with a gas manifold to route purified inert gases or inert-hydrogen gas mixtures through the pots. A typical setup with two pot/heater assemblies is shown in Figure 2.

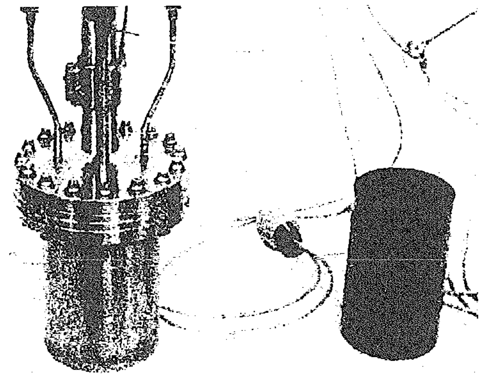

Figure 1. Pot and crucible for Flibe preparation.

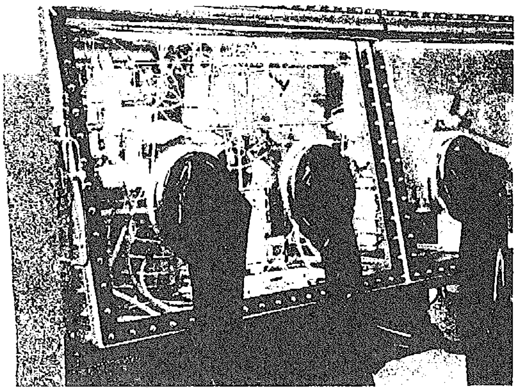

Figure 2. Glovebox setup with two pot/heater assemblies and external cooling jackets.

We have incorporated temperature sensors and an on-line quadrupole mass spectrometer (QMS) to monitor key parameters of the thermo-cycle process during heat-up and cool-down of the salt mixture. A temperature sensor is inserted into a Ni sheath tube in the salt mixture and another is used to control the pot heater. The on-line QMS is used to measure the gas composition of the process stream that is flowing through the pot during the thermo-cycle. This analysis provides a useful measure of off-gassing from the salt components before melting and during the melt phase. An example of the data measured for an initial thermo-cycle of Flibe components is shown in Figure 3. The pot was heated on a step profile, with hold temperatures of 200, 400 and 800°C during the heat-up. For this cycle, the Flibe components were not individually degassed (the usual process) so we observed a significant increase in the QMS mass-18 signal for pot temperatures less than 400°C, indicative of water release from the powder material. As the pot temperature rose through 600°C, the QMS mass-18 peak was unstable but showed indications of additional water release as the $BeF_2$ melted. During the cool-down, there was a temperature arrest at a measured temperature of 459°C, indicative of the liquid to solid phase transition for Flibe.

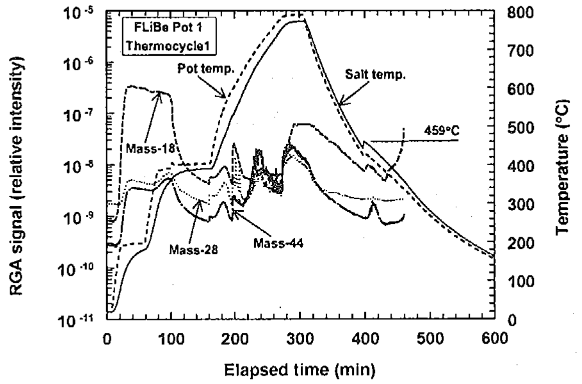

Figure 3. Thermo-cycle data for initial heat-up of Flibe components and cool-down of Flibe sample.

The on-line QMS was used in experiments to measure gas release from molten Flibe pots in an evaluation of He-bubble purge effectiveness and purge gas pot material interactions. Bubbling through the Flibe with high-purity helium enhanced water release from the salt, and the release rate of water and HF increased with temperature. Experiments were conducted with purge gases consisting of He and $H_2$ or $D_2$ to investigate isotope exchange reactions in the Flibe pot environment and purge gas interactions with pot/Flibe materials [14]. As shown in Figure 4 for tests at 600°C, hydrogen interactions with the stainless steel pot only were minimal. However, hydrogen interactions were more pronounced when the pot contained a blank carbon crucible and they were especially pronounced when the pot contained a carbon crucible with Flibe. These tests demonstrated that even though carbon is suitable for many operations with molten Flibe, the potential for hydrocarbon generation could complicate the interpretation of planned tritium behavior studies in Flibe. As a result, we selected Ni as the Flibe-facing material for tritium behavior studies.

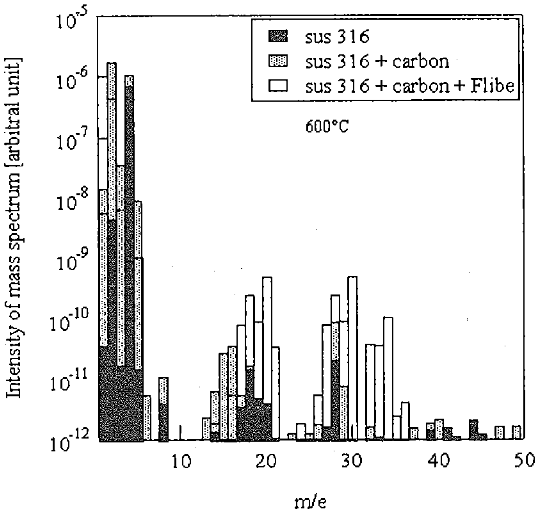

Figure 4. Comparison of QMS mass spectra for experiments in environments with (a) type 316 stainless steel pot, (b) stainless steel pot and carbon crucible and (c) stainless steel pot, carbon crucible and Flibe.

A hydro-fluorination process, based on the early work on the MSRE [15], is used to purify Flibe by reducing metal oxide impurities and sulfur-bearing compounds. Our implementation of this technique employed purging of the molten Flibe at a nominal temperature of 520°C for several days with a mixture of He, $H_2$ and HF in which the $H_2$ concentration is at least 10 times the HF concentration. During the purification process, the HF concentrations in the gas mixture flowing through the molten salt are measured using titration techniques. Comparison of HF concentrations in the purge line downstream from the Flibe pot with those in the upstream gas provided an indication of the progress in oxide reduction interactions in the salt. For example, at the start of the purification run, HF concentration downstream is much less than upstream, and as the oxide content in the salt is reduced, the downstream HF concentration rises to approach the upstream value. Currently, a Schott auto-titrator is implemented to provide on-line measurements of the pH in a titration cell that the gas stream is bubbled through.

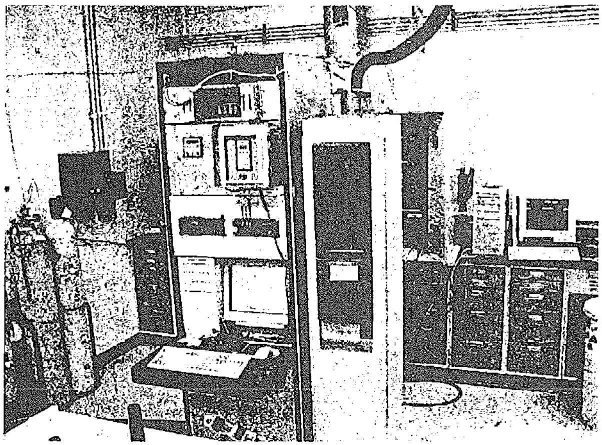

Figure 5. Control instrumentation and gas cabinet for He-HF gas cylinders.

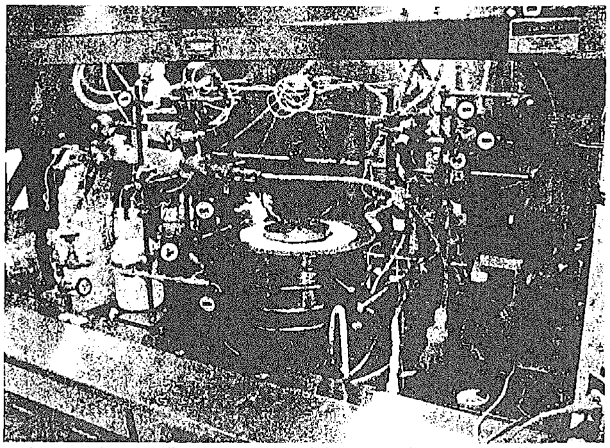

Figure 6. Pot/heater assembly, HF titration cell, gas manifolds and HF traps for purification setup.

A variety of analytical techniques are used to assay the impurity content of the chemical reagent feedstock for Flibe and the impurity content of the Flibe before and after the hydro-fluorination purification. Oxygen, nitrogen, and carbon were determined by methods using LECO® equipment. Metals analyses were by measurements using inductively-coupled-plasma atomic emission spectroscopy (ICP-AES) and ICP-MS (mass spectroscopy). Results of these analyses for a representative batch of Flibe and the feedstock ingredients are given in Table 1. Noting that $BeF_2$ comprises 1/3 of a molar fraction of Flibe, these results show that the hydro-fluorination process has reduced the oxide content by a factor of 3 in the processed material. Additional hydro-fluorination processing can reduce the oxide content further.

Table 1. Measured impurities in Flibe chemical components and purified Flibe

| | O (ppm) | C (ppm) | N (ppm) | Fe (ppm) | Ni (ppm) | Cr (ppm) |
|---|---|---|---|---|---|---|
| $BeF_2$ | 5700 | <20 | 58 | 295 | 20 | 18 |
| LiF | 60 | <20 | 78 | 100 | 30 | 4 |
| Flibe | 560 | 10 | 32 | 260 | 15 | 16 |

## 3. REDOX CONTROL

Our current strategy is to test the effectiveness of beryllium metal as a REDOX control agent in a Flibe system that contains free fluorine and TF. The primary basis for this choice is two-fold: (1) the free-energies of formation for elements of importance to a Flibe system (see Table 2), indicate that $BeF_2$ is more stable than most metal fluorides and TF, and (2) Be is needed for neutron multiplication in the Flibe blanket to achieve a suitable tritium breeding ratio.

Table 2. Free energies of formation at 1000°C

| Fluoride | Free energy (kcal/g-atom of F) |
|---|---|
| $MoF_6(g)$ | -50.84 |
| $WF_6(g)$ | -56.84 |
| $NiF_2(s)$ | -60.02 |
| HF | -66.55 |
| $FeF_2(s)$ | -67.9 |
| $CrF_2(s)$ | -77.04 |
| $MnF_2(s)$ | -85.27 |
| $BeF_2(l)$ | -104.45 |
| $LiF(s)$ | -124.12 |

Previous studies have indicated that the use of metallic Be in Flibe does significantly reduce corrosion of type 316 stainless steel [16] and does facilitate the conversion of TF to HT in neutron-irradiated Flibe [17]. However, these experiments do not provide sufficient information regarding the kinetic behavior of the Be reactions with free fluorine ($F_2$) and/or fluoride (HF) to produce $BeF_2$. One of the initial goals of our research is to elucidate the kinetic behavior of Be in Flibe. This will entail measurements of the reaction rates of the relevant processes and the determination of the most effective physical form for the REDOX agent (e.g. dissolved vs solid surface or coating).

Studies have been initiated to investigate the reaction kinetic behavior of solid Be surfaces with known concentrations of HF in Flibe. These experiments are based on an adaptation of the purification system and a REDOX reactor illustrated in Figure 7. The approach uses a pot-configuration similar to that for the purification experiments with the adaptation to provide for controlled insertion of a Be foil or solid cylinder in the molten salt. The initial approach consists of: (1) purging the salt with He-HF (HF concentrations of about 1000 ppm) to establish an equilibrium concentration of HF in the salt, (2) stopping the HF purge while retaining a pure He purge, (3) inserting a Be rod into the molten salt, (4) all the while, monitoring the HF concentration in the outlet gas stream with the on-line auto-titrator.

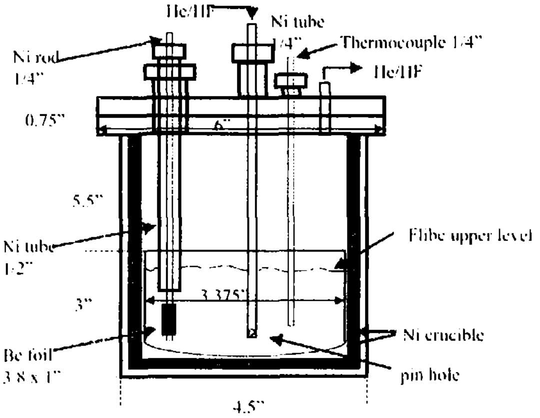

Figure 7. Illustration of REDOX reactor.

In principle, changes in the outlet stream HF concentrations can be related to various processes influencing HF transport from the pot environment (e.g., reduction in HF concentrations in the pot head-space due to volumetric sweep-out with only He, HF transport across the Flibe/gas-phase interface, reactions of Be with HF in solution in the salt and a consequent change in the rate of HF transport across the liquid/gas interface). Current tests are underway to establish the ultimate detection sensitivity of the auto-titrator for this application.

## 4. TRITIUM BEHAVIOR IN FLIBE

The ultimate goal of this task is to characterize tritium behavior in Be-REDOX controlled Flibe. We know that interrelated transport processes and chemical interactions characterize the behavior of hydrogen isotopes in molten fluoride salts, but the available results do not provide sufficient information to reliably quantify tritium behavior in a REDOX-controlled Flibe blanket system. Important results concerning tritium species in neutron-irradiated Flibe have been reported previously [17] and references therein. These studies indicated that tritium was primarily present as TF in irradiated Flibe, unless the molten Flibe was exposed to an overpressure of $H_2$ resulting in an increase in the solution hydrogen that facilitated the change from TF to HT. However, for a Flibe Blanket, use of hydrogen complicates and adds a significant burden to the tritium recovery and isotopic enrichment plant. Furthermore, as stated in Section 1, the transport data for diffusion and solubility are derived from a very limited data set.

Because of this history, we proceeded on a plan to investigate the deuterium transport properties in purified Flibe before we initiated work with tritium or proceeded to Be-REDOX-controlled Flibe. This research entailed the development of a dual permeation probe system that could be accurately modeled and used in experiments to evaluate diffusion and solution properties of hydrogen isotopes in Flibe. Initial work in this development has been reported previously [14, 18] and more detailed updates of this work are presented at this workshop [19] and at the ICFRM-11 meeting [20]. A brief summary of this work is included here.

The basis of our experimental and analytical approach is illustrated in Figure 8. The heart of the experiment is a cylindrically-symmetric, dual probe permeation pot system that consists of a type-316 stainless-steel pot, a nickel crucible that contains about 400-cc of Flibe, two permeation probes of thin-walled nickel, a gas manifold to provide Ar purge gas flow through assembly volumes, and a quadrupole mass spectrometer for on-line measurements of the flow-stream gas compositions. Typically, with the pot at a test temperature, probe-1 is pressurized with deuterium to about 0.9 atm and QMS analysis of Ar gas purging through probe-2 provides a measure of the deuterium that has permeated through probe-1 wall, the molten Flibe between the probe walls and through the wall of probe-2. Figure 9 shows the pot/heater assembly and associated gas-flow lines that are installed inside a glovebox that is operated with an inert gas environment during experiments.

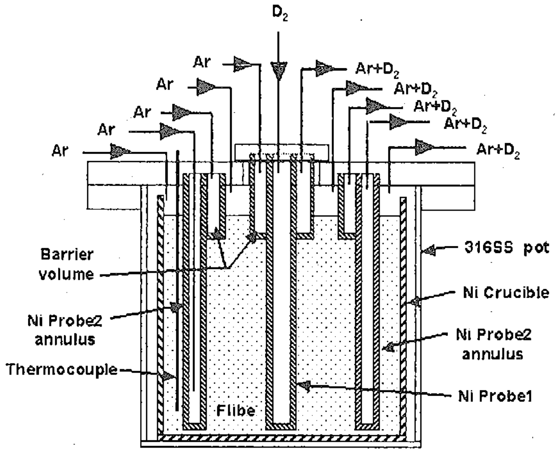

Figure 8. Schematic illustration of cylindrically symmetric, permeation probe assembly.

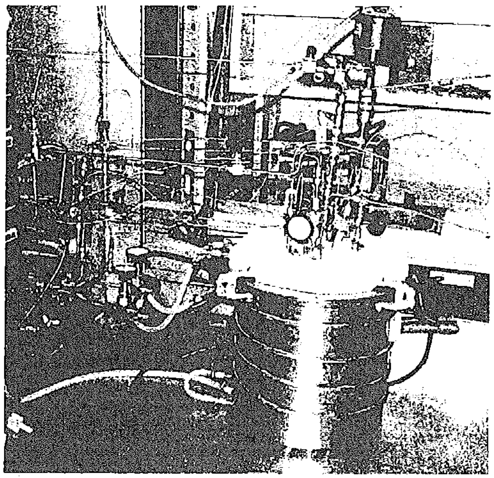

Figure 9. Glovebox setup for permeation probe exp.

Detailed model simulation calculations have been done for this experimental system [14,18,19] with and without Flibe. Permeation experiments without Flibe were done to demonstrate the performance of the gas handling system and the on-line QMS analysis approach and to benchmark the model calculations against measured deuterium permeation data. The analysis indicated excellent agreement with previously published data for hydrogen transport in nickel [14,18]. Systematic simulation calculations for the Ni/Flibe/$D_2$ system demonstrated that permeation through Flibe was rate-limiting for conditions in our experiments. This result is demonstrated in Figure 10 that compares measured deuterium transport data for experiments at 600°C without Flibe (Exp A) and with Flibe (Exp B) for comparable Ar purge flow rates through probe-2. Two features of the Exp B data reflect the influence of Flibe on permeation in the Flibe. First, there is significant time delay in the probe-2 $D_2$ permeation signal and in the buildup of deuterium above the Flibe. Second, the maximum $D_2$ partial pressures in probe 2 and above the salt are much less than those observed in Exp A. These results are due to the low solubility and slow diffusivity of $D_2$ in Flibe as predicted by the simulation calculations.

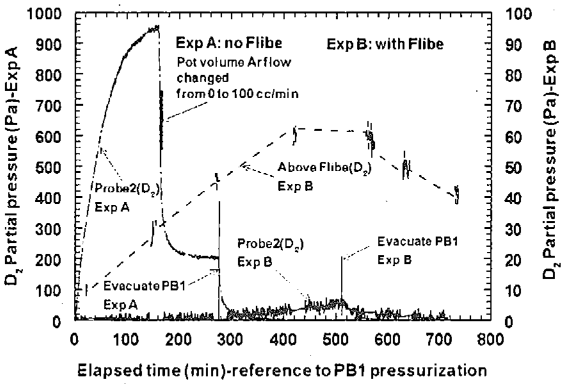

Figure 10. Comparison of measured deuterium data for experiments with and without Flibe.

Diffusion coefficients were derived from a cylindrical model analysis of measured permeation rates for experiments at 600 and 650°C. Details of this analysis are reported elsewhere [19, 20]. Results from these analyses are compared in Figure 11 with previously published data for hydrogen isotope diffusion in Flibe. Our diffusion results are a little less than those reported by Oishi et al. [21] for $T^+$ transport in molten Flibe, suggesting that deuterium was also diffusing as the $D^+$ species in our experiments. Both of these results are somewhat greater than those derived from the viscosity using the Stokes-Einstein relation, $D = kT/(6\pi R\eta)$, assuming a particle radius, R, of 2 Angstroms and a viscosity, $\eta$, reported by Cantor [22] for $2LiF \bullet BeF_2$. The diffusion activation energy for the Oishi et al. data is consistent with the viscosity data. However, a two-point fit to our diffusion coefficients (a dangerous approach with such limited data) yields a diffusion activation energy comparable to that for $F^-$ self diffusion reported by Ohmichi et al. [23]. Such a result would suggest transport mechanisms similar to that postulated by Ohmichi et al., namely ion-pair diffusion or exchange processes that break Be-F bonds. Data are also shown in Figure 11 for diffusion of $T^+$ in solid Flibe. These diffusion results, along with high values of solubility derived for our permeation probe experiments [19,20] suggest that deuterium transport in our experiments was mediated by the presence of a bond between $D^+$ and $T^-$ in the molten salt.

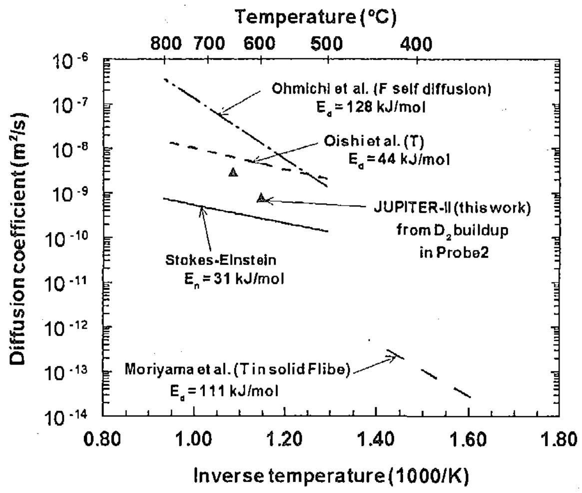

Figure 11. Diffusion data for Flibe.

## 5. SAFETY TESTS (FLIBE MOBILIZATION)

A safety concern for a Flibe breeder blanket is the potential mobilization of tritium and Flibe vapors and aerosols due to an accidental breach of the Flibe containment structure. To address the issue of Flibe mobilization, we have conducted a systematic series of tests in which molten Flibe was exposed to argon, air and moist air at temperatures from 500 to 800° and mobilization rates and products were characterized. Detailed results of this work were reported at ICFRM-11 [25]. A summary of key features of this work is reported here.

Mobilization tests were performed in a transpiration setup similar to that described by Sense et al. [26] for vapor pressure measurements. A schematic of our test system is shown in Figure 12. The system consisted of the following: (1) an inlet gas manifold for introducing Ar air or moist air, (2) a gas pre-heater, (3) a Ni tube exposure chamber centered inside a tube furnace,(4) a thermocouple for test temperature measurements at the sample location, (5) an Inconel-600 vapor-transfer tube (probe) that is inserted inside the exposure chamber with its entrance tip positioned near the sample, (6) a collection tube that is directly connected to the probe, (7) a condenser for moisture removal during moist air experiments, and (8) an exhaust line to a hood. Samples were tested in either nickel or glassy carbon crucibles, dependent on the exposure conditions. A quadrupole mass spectrometer was used to verify leak tightness of the system prior to initiation of an experiment.

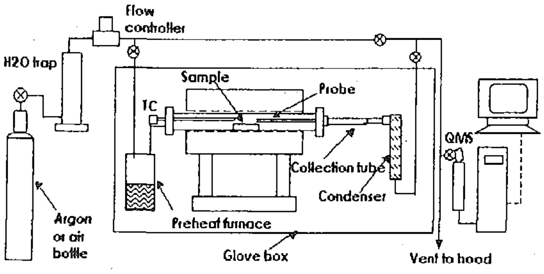

Figure 12. Illustration of transpiration setup for Flibe mobilization experiments.

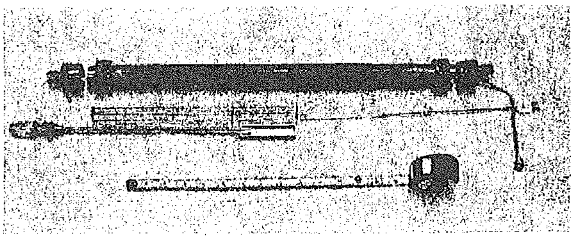

Figure 13. Nickel test assembly for Flibe mobilization experiments

Figure 13 shows the components of the nickel assembly that are installed in the tube furnace for each test. The exposure chamber is at the top, the probe tube, nickel sample crucible, and thermocouple are located in the center of the photo. The setup of the mobilization system, as it is configured inside an inert-gas glovebox, is shown in Figure 14.

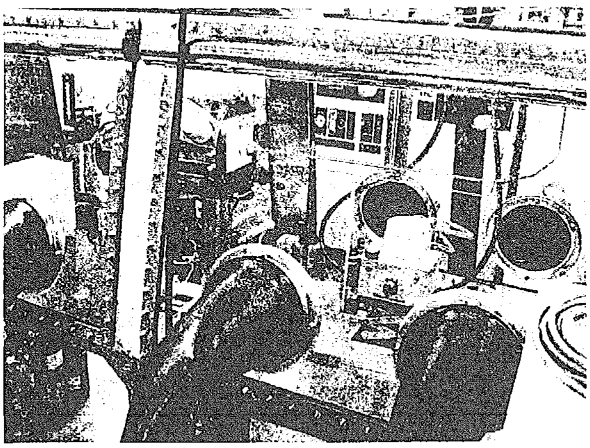

Figure 14. Setup of Flibe mobilization experiment inside inert gas glovebox.

Determination of the quantity and chemical species of material transported required removal of the probe and stainless steel collection tube. The internal surface of the stainless steel tube and the inside and outside surfaces of the probe tube were separately rinsed with 10% nitric acid and the solutions were analyzed for Be and Li by ICP-AES. Measurements from the internal probe surfaces were used for vapor pressure calculations. Deposits from the external probe surfaces were used to compare the amounts and ratios of Be and Li on internal and external surfaces. The Ni exposure chamber was removed and cleaned after selected test series.

Both Ni and glassy carbon crucibles were used in these experiments. The Ni was suitable for experiments in pure Ar but unsuitable for use in air or moist air because it readily oxidized in the air. This created a surface that was more easily wetted by the Flibe enabling it to flow out of the crucible. Glassy carbon crucibles provided good containment for Flibe under air and moist-air exposure conditions but experienced mass loss because of oxidation. Sample mass loss measurements were made for tests with Ni crucibles in Ar to evaluate mass balance. Such measurements were not useful for experiments with glassy carbon crucibles in air.

Initial mobilization tests were performed with pure LiF at 875 and 1000°C to validate the experimental approach and system. Vapor pressures derived from these experiments were in reasonable agreement with results reported by Sense et al. [27] indicating that our test geometry and flow rates provided reasonable conditions to maintain saturation above the molten material.

Mobilization tests were performed with Flibe that was previously purified as described in Section 2 of this paper. Tests in Ar and air were conducted at 500, 600, 700 and 800° with flow rates of 25-100 sccm. Tests in moist air were done at 600 to 800°C at flow rates of 25 and 50 sccm.

Partial vapor pressures for $BeF_2$ and $LiBeF_3$ and the resultant total vapor pressures (sum of the partials) were derived from the experiments for Flibe in a pure Ar environment. Results are plotted in Figure 15, along with values from high temperature transpiration experiments (ORNL data(1) [28]), from extrapolations of ORNL data (ORNL extrapolation), from calculated vapor pressures based on thermodynamic models and derived activity coefficients [29], and from mass spectrometric measurements of the vapor species ($BeF_2$ and $LiBeF_3$) above Flibe at 600°C [30]. The INEEL results were in excellent agreement with the mass spectrometric data but were a factor of 2-3 below the extrapolated or computed data. The INEEL results also showed increasing contribution from Li-bearing species at the higher temperatures, a result consistent with the mass spectrometric species determination.

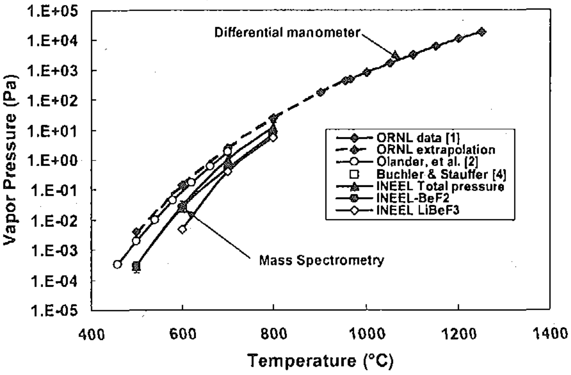

Figure 15. Total vapor pressure over Flibe.

Vapor pressure results for $BeF_2$ derived from the experiments with Ar, air and moist air are compared in Figure 16. These results show that there was no significant influence of these environments on mobilization behavior.

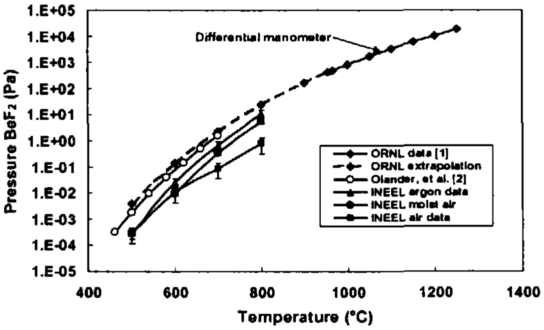

Figure 16. $BeF_2$ vapor pressure over Flibe exposed to different environments.

## 6. SUMMARY

This paper presented an overview of Flibe molten salt research activities concerning that were conducted at the INEEL as part of the JUPITER-II joint research program. The focus of these efforts addressed tritium/chemistry and safety issues relevant to use of Flibe in a tritium breeder. Progress has been made in four areas: (1) Flibe preparation, (2) REDOX control, (3) Flibe tritium behavior, and (4) Safety studies.

We have developed the process systems and capabilities to produce close to liter-size quantities of Flibe and purify them (reduce oxide impurity content) using the hydro-fluorination technique. Sufficient material for initial test requirements has been made and analyzed using standard chemical analyses tools. This work has been largely done in inert-gas glovebox systems to maintain environmental integrity during material handling operations and prevent the spread of Be particulate to the worker environment.

REDOX experiments have been initiated using the system developed for molten salt purification. These experiments hinge on the use of a sensitive on-line auto-titrator to measure small changes of HF concentrations in a process stream flowing through a REDOX test vessel. This vessel is patterned after our purification pot assemblies and includes the capability to insert a metallic Be sample into the molten salt. Planned experiments will evaluate the system for kinetic behavior of Be reactions with HF.

An initial set of permeation experiments has been completed in an effort to better understand and quantify deuterium diffusion and solubility behavior in molten Flibe. These experiments were done with a uniquely designed, double permeation probe assembly that was accurately modeled with TMAP-4 [31], a one-dimensional computer simulation code, and with a model representation in cylindrical coordinates. Diffusion and solubility coefficients derived from these experiments indicated that deuterium transport in the Flibe experiments was most likely mediated by the presence of a bond between $D^+$ and $T^-$ in the molten salt.

To address safety issues, we have developed process systems and procedures that enable safe handling of Be-bearing compounds, and we have conducted a series of experiments to measure mobilization of vapors and aerosols from Flibe exposed to Ar, air, and moist air environments. Vapor pressures derived from the mobilization experiments were in excellent agreement with previous mass-spectrometric measurements at 600°C but were a factor of 2-3 below values based on recent calculations and extrapolation of high-temperature transpiration data. Our measurements showed increasing concentrations of Li in the mobilized fraction as the temperature was increased to 800°C. The results showed that there was no significant influence of the differing environments (Ar, air, moist air) on mobilization behavior.

Future research will focus on the following: (1) continued development/implementation of improved Flibe purification techniques, (2) development and testing of a REDOX control strategy using Be and (3) investigation of tritium behavior and control in a REDOX-controlled Flibe. This work is directed toward ultimate integrated long-term corrosion tests for Flibe breeder blanket materials.

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