MASTER

COO-4262-5

LITHIUM OXIDE
MICROSPHERE FABRICATION

Monthly Letter Progress Report #5
for the Period Ending September 30, 1977

Prepared by

T. A. Thornton
The Babcock & Wilcox Company
Lynchburg Research Center
Lynchburg, Virginia 24505

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Submitted by

The Babcock & Wilcox Company
Contract Research Division
P. O. Box 835
Alliance, Ohio

950 6546

Date Published — November 1977

Prepared for the United States Energy Research & Development Administration
ERDA Contract EG-77-C-02-4262
B&W Contract No. CRD 1007

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

eb

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

"This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States ERDA, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights."

TASK 1 - Lithium Oxide Microsphere Fabrication and Analysis

1.1 Introduction

1.1.1 The purpose of this task was to establish basic process requirements and techniques for the fabrication of lithium oxide (Li_2O) microspheres with properties which as closely as possible approximate those required by the design characteristics of the University of Wisconsin reference TOKAMAK fusion reactor⁽¹⁾. This reactor would involve the utilization of a gravity flow bed of lithium oxide microspheres for the purposes of tritium breeding (via neutron capture in Li), heat transport, and steam generation. As a basis for microsphere fabrication studies, the requirements are for pure lithium oxide microspheres with the following general properties:

- a. Approximately 100 μm microsphere diameter,
- b. Approximately 85 percent microsphere theoretical density,
- c. Approximately 1g/cc bed density
- d. Average microsphere open pore-to-pore spacing $\sim 1 \mu\text{m}$,
- e. Microsphere strength sufficient to withstand significant attrition rates in the proposed reactor particle transport system.

These microsphere requirement guidelines were regarded as only very general and giving considerable leeway in the development of fabrication processes.

1.1.2 The research conducted at the Babcock & Wilcox Lynchburg Research Center concerning the specified lithium oxide microspheres may be divided into the following general categories;

- a. Study of basic lithium oxide chemistry,
- b. Conceptual and experimental studies of candidate processes for the production of lithium oxide microspheres,
- c. Production of small quantities of microspheres by those processes which show promise as a basis for commercial production.
- d. Analysis of these microspheres for density, porosity, and chemical purity characteristics.

- e. Rough estimation of costs for large scale production of microspheres.
- f. Identification of future R&D requirements in the area of microsphere fabrication techniques.

This section is written as a summary of the research conducted in these areas under Task 1 of the research effort.

1.2 Lithium Oxide Chemistry

1.2.1 In order to serve as a baseline for the analysis and evaluation of lithium oxide microsphere fabrication processes, a literature search and some experimental work were conducted in the area of basic lithium oxide chemistry. The literature search primarily involved a review of the material available in Chemical Abstracts from 1930 to the present. Also involved were discussions with personnel of the University of Wisconsin Chemistry Department and observations of experimental work performed there in the generation of lithium oxide powders by the peroxide precipitation technique.

This study of lithium oxide chemistry was conducted from two general standpoints; methods for the generation of pure lithium oxide starting material for microsphere fabrication processes, and chemical reactions involving lithium oxide which affect the fabrication process or the microsphere moving bed reactor blanket system.

1.2.2 There are several ways to make lithium oxide. It can be prepared by:

Pyrolysis (thermal decomposition) of the hydroxide, carbonate, or peroxide

Combination of the elements, either directly,

or indirectly by reaction in liquid ammonia

None of these processes yields a pure product in straightforward fashion. Reactions (1.1 to 1.3) take place at high temperatures with large heats of activation. They are best carried out in high vacuum systems. The reaction products therefore contain variable amounts of impurities including both undecomposed starting materials and materials picked up by interaction with the reaction vessel. Of these three processes (1.3) seems to offer the best chance for a pure product for two reasons: There is no strong reverse reaction and it takes place at lower temperatures. Thus "complete" decomposition is easier to obtain and the lower temperature makes crucible reactions less likely. On the other hand, the peroxide releases pure oxygen, the most hostile of the gaseous products of any of the first three reactions. In addition most procedures found in the literature⁽²⁾⁽³⁾⁽⁴⁾⁽⁵⁾⁽⁶⁾ use organics in the preparation of the peroxide. Traces of residual organics are virtually certain to convert to carbon dioxide which will be partly picked up by the oxide.

Reactions (1.4) and (1.5) offer their own complications. In its reaction with free oxygen lithium metal produces some peroxide as a byproduct. Thus the direct synthesis leads back to some of the same complications offered by reaction (1.3). Moreover the reaction is hazardous. It is extremely slow at room temperature in dry oxygen and must be heated to get started. Once started, it can easily run away. Reaction (1.5) runs smoothly at room temperature. Lithium dissolves in liquid ammonia to form the same kind of well-known blue solvated system which forms with sodium. Admission of oxygen produces oxide with a low peroxide content. The oxide is also free of moisture or carbon compounds but will contain small amounts of the amide convert on firing to the nitrite.

In the present study of lithium oxide microsphere fabrication, the lithium oxide preparation reactions of interest are primarily those shown in equations (1.2) and (1.3). Experimental work was performed utilizing off-the-shelf -325 mesh, ~96% pure, lithium oxide powder and lithium peroxide generated at the Lynchburg Research Center. The purchased lithium oxide powder was produced via the lithium carbonate decomposition reaction, which is the primary commercial source of lithium oxide⁽⁶⁾. Details of the lithium peroxide preparation process will be presented later in this report.

Of particular note regarding the experimental work, however, is the fact that neither the purchased lithium oxide powder nor the lithium peroxide generated in the laboratory is very pure. Analyses of the as-received lithium oxide powder reveal, by titration analysis, a purity range of 94 to 96%, with the impurity balance consisting primarily of lithium hydroxide and lithium carbonate. The lithium peroxide which is generated was found, by permanganate titration analysis, to be as low as 91% pure lithium peroxide (Li_2O_2), with lithium hydroxide the principal impurity. The presence of lithium hydroxide (LiOH) and lithium carbonate (Li_2CO_3) as impurities in lithium oxide microspheres compromises the required physical properties of the microspheres in a manner which will be discussed later in this section.

Table 1-1 presents a short summary of some of the physical and chemical properties of lithium oxide of importance to the present research.

Table 1-1 - Properties of Lithium Oxide

Melting Point - variously given as 1843°K, 1700°K, 1973°K, 2000°K

Boiling Point - variously given as 2873°K, 2836°K, 2600°K

Molecular Weight - 29.88 grams/mole (46.44% by weight Li)

Density - 2.013 grams/cc

Heat of Formation - 142.9 kcal/mole (298°K)

Entropy of Formation - 9.06 cal/mole (298°K)

Free Energy of Formation - 134.3 kcal/mole (298°K)

Heat of Fusion - 14 kcal/mole

Heat of Evaporation - 56 kcal/mole

Heat of Sublimation - 110 kcal/mole

Specific Heat - 12.9 cal/mole - °K

Vapor Pressure (torr) -

	<u>1200°K</u>	<u>1500°K</u>	<u>1773°K</u>	<u>2000°K</u>
JANAF	4.17×10^{-7}	1.51×10^{-3}	.209	4.19
Russian Data	3.6×10^{-8}	2.3×10^{-4}	.044	.64

1.2.3 The chemical reactions of primary interest to this research project which lithium oxide undergoes are the reverses of equations (1.1) and (1.2). Lithium oxide reacts readily and exothermically with water and carbon dioxide to form, respectively, lithium hydroxide and lithium carbonate. The primary difficulty encountered in the microsphere fabrication processes evaluated in this study was the prevention of the formation of substantial quantities of the hydroxide and carbonate during processing. The presence of hydroxide and carbonate in the finished lithium oxide microsphere product reduces the melting and sintering points of the microspheres. Although the literature search produced no available data on the phase relationships of the Li_2O - LiOH - Li_2CO_3 ternary system, some data, presented in Figures 1-1A and 1-1B, does show phase relationships for the Li_2O - Li_2CO_3 binary system as determined by two separate researchers⁽⁷⁾⁽⁸⁾. Of primary interest in these two figures is the disparity in the presented data. In particular, the location of the eutectic point at nearly opposite extremes of the Li_2O - Li_2CO_3 ordinate is noteworthy. This conflict in published phase equilibrium data is unfortunate in that the effect of small amounts of lithium carbonate impurity in the fabricated lithium oxide microspheres could heavily influence the properties of the microsphere bed. Additionally, furnace sintering tests on some experimentally produced microspheres show considerably decreased sintering/melting points of the microspheres with comparatively small ($\sim 15\%$) lithium hydroxide levels. This data will be discussed later in this section.

From the standpoint of materials used in microsphere fabrication equipment, both experimental and commercial, two more reactions of lithium oxide might here be mentioned. Lithium oxide can react with silica⁽⁹⁾⁽¹⁰⁾ (SiO_2) at temperatures from 800 - 900°C to form lithium metasilicate (Li_2SiO_3), and can further react at higher ($\sim 1250^\circ\text{C}$) temperature to form orthosilicates, Li_4SiO_4 , Li_6SiO_5 , and Li_8SiO_6 . Lithium oxide also reacts with alumina (Al_2O_3) to form the lithium aluminates, LiAlO_2 and Li_3AlO_3 , at approximately 1350°C . These reactions are not presently perceived as problems since none of the examined fabrication methods involve direct contact of the lithium oxide/peroxide/hydroxide materials with pyrex, glass, aluminum, or other such materials at these temperatures. However, the evaluation of any candidate microsphere fabrication processes should account for possible lithium oxide reactions with such materials as silicates, borates, aluminates, ferrates, etc.

FIGURE 1-1A: Li_2CO_3 - Li_2O PHASE EQUILIBRIUM DATA OF PAPIN ET AL (7)

FIGURE 1-1B: Li_2CO_3 - Li_2O PHASE EQUILIBRIUM DATA OF SMIRNOV ET AL (8)

1.3 Evaluation of Microsphere Fabrication Processes

1.3.1 Figure 1-2 shows a general flow chart for the microsphere fabrication processes considered as a part of the research effort. These processes generally involve the sequence of lithium oxide/peroxide powder preparation, granulation of the powder to the approximate desired size and free-flowing character, screening for particle recycle, sintering/firing to a dense microsphere, and particle analysis. Most of the detailed processing methods considered for each of these steps had the advantages of conceptual, mechanical, and chemical simplicity. For convenience, the processing steps will be presented for evaluation in the categories of preparation, pregranulation, and firing.

1.3.2 A number of processing methods were studied experimentally and conceptually, and evaluated primarily from these criteria; the chemical purity of the product, the size and size distribution of the particles, and the free flowing quality of the bulk particles. Other criteria were the simplicity, and the chemical handling and safety characteristics of the operation. The following is a brief summary of each process considered and an evaluation of their feasibility based on the above mentioned criteria.

1.3.2.1 Powder Preparation

Two substances were used and evaluated as starting materials for lithium oxide microsphere fabrication; -325 mesh, ~95% pure lithium oxide purchased from a chemical specialty retailer, and lithium hydroxide monohydrate powder obtained in the same manner.

The non-free-flowing, -325 mesh lithium oxide powder was used directly as feed material for the pregranulation methods subsequently explained in this section. This material was ultimately supplied by the Lithium Corporation of America, where it was prepared by the decomposition of lithium carbonate obtained from naturally occurring lithium bearing minerals.⁽⁶⁾

The lithium hydroxide monohydrate was used as the starting material for the precipitation of lithium peroxide. The detailed procedures for this precipitation method are given in Appendix A. In summary, the procedure involves the precipitation of lithium peroxide from aqueous lithium hydroxide by the addition of 30% hydrogen peroxide. It was found at the University of Wisconsin that a fairly pure (96-98%) lithium oxide powder could be generated

FIGURE 1-2 GENERAL MICROSPHERE FABRICATION FLOWSHEET

by the calcination of the lithium peroxide formed by this method. For the purpose of attempting to fabricate lithium oxide microspheres, however, this lithium peroxide powder was not bulk calcined, but, being reasonably free flowing as the dried precipitate, was directly used in the firing/sintering steps subsequently explained here. Both these powder preparation steps were thus eventually used to generate microsphere samples.

1.3.2.2 Powder Pregranulation

A number of methods to accomplish the pregranulation of the -325 mesh lithium oxide powder were studied. These methods can generally be divided into dry mechanical and wet, or sol, chemistry methods.

The dry mechanical methods examined included:

a. Fluidized Bed Agglomeration

Experiments in the fluidized bed agglomeration of the lithium oxide powder were conducted by B&W personnel at Glatt Air Techniques, a fluidized bed development laboratory. The fluidized bed agglomeration technique involves the gas fluidization of a bed of the lithium oxide powder concurrent with the spray injection on the bed of fine droplets of a binder material (in this case ethylcellulose, KLUCEL, or ethyl silicate dissolved in acetone).

It was found that this technique successfully produced a well-sized agglomerated material which demonstrated excellent free-flowing properties. However, the fluidized bed unit used in the experiment did not have the capability to adequately dry the air used as fluidizing gas, with the result that the agglomerated material was considerably contaminated with lithium hydroxide and lithium carbonate formed from the reaction of the lithium oxide with the residual water vapor and carbon dioxide in the fluidizing air. Also, some difficulty with the lithium oxide fines dispersing in the laboratory air during transfer of the powder to and from the unit was experienced. The process did show enough promise, however, to justify this pregranulation method for further study.

b. High Intensity Mixing

Experiments were conducted in the dry mixing/agglomeration of lithium oxide powders using a LODIGE M-5-G laboratory mixer. In this method a batch of the lithium oxide material would be agglomerated to a desired granulate size by mixing the material with a spray of the same binder materials

as used in the fluidized bed agglomeration. However, it was found that such parameters as mixing speed and mixing time could not be controlled adequately enough to granulate the very light lithium oxide powder. Additionally, the lithium oxide powder could not be satisfactorily transferred to and from the mixer without releasing considerable amounts of the noxious lithium oxide fines into the surrounding air. This combination of inadequate fines handling and poor progranulation characteristics eliminated this method from further consideration.

c. Pellet Pressing/Recrushing

Experiments were conducted which involved the die pressing of the lithium oxide powder into pellets, followed by the sieve crushing of the pellets to form a free flowing granulate. At certain pellet press pressures, this method demonstrated a capability to produce a slightly free flowing agglomerate suitable for flame injection firing. Variables which can be altered in this method include pressing strength, kind and amount of dry blended binder added, and sieve crushing size. Disadvantages of this method seem to be barely adequate size distribution of the agglomerated material, and the imperfectly free-flowing quality of the agglomerate.

Wet chemical methods for the fabrication of a sized, free-flowing lithium compound pregranulate which were considered in the research were:

1. Sol-Gel Spheroidation

Experiments were conducted to determine whether a feasible method existed for the production of gelled microspheres of lithium oxide, starting from the purchased, -325 mesh, lithium oxide powder. Sol-gel spheroidation involves the formation of a stable suspension, or sol, of the material to be spheroidized, usually in an aqueous system, followed by the countercurrent extraction of the water from droplets of the sol by a liquid dessicating agent.

It was found that the formation of an aqueous sol of lithium oxide, even in an extremely high pH solution, was impossible due to the highly dominant, hydroxide forming, reverse reaction of equation 1.1. The reaction of the lithium oxide to form lithium hydroxide in an aqueous medium always goes to completion. Attempted formation of lithium oxide sols in light alcohol media gave the same results. There was some indication that a stable

sol of lithium oxide in acetone may be possible, but the lack of a known extraction agent for the acetone, and the lack of known stabilizing agents for this system precluded further consideration.

Due to these considerations, the sol-gel method for the formation of lithium oxide microspheres was not considered further in this study.

2. Spray Drying

Due to essentially the same considerations given above, the study of a possible spray drying method for the formation of free flowing sized granules of lithium oxide was confined to the lithium oxide/acetone system. Small scale testing of this method using slurries of lithium oxide in acetone sprayed through Babington Nebullizers and simple spray nozzles were unsuccessful. The primary reasons for the rejection of this method for further consideration in this study were the lack of slurry stability, and safety and toxicity considerations. The unstable nature of the slurry caused repeated clogging of nozzles and flow passages in the equipment used. This problem might be alleviated if a slurry stabilizing agent could be found for the Li_2O /acetone system, but this agent was not found among the few surfactants examined (Darvan #7, Kodak Photo-Flo, Vahzak 112, Darvan DC 772) and was not further pursued. It was also found that the construction of a safe lab-scale spray drying apparatus for this application (due to the extreme volatility of acetone) was beyond the scope of this study. The results of the Babington Nebullizer and simple spray nozzle studies also demonstrated an excessive generation of extremely fine droplets which presented Li_2O toxicity problems inside the laboratory. For these reasons further investigation of the spray drying technique was eliminated.

3. Lithium Peroxide Precipitation

This method for producing a free flowing, sized lithium peroxide particle as a feed for the sintering/firing steps has already been discussed in section 1.2. It should only be noted here that this method involves the production of lithium peroxide rather than lithium oxide for subsequent firing.

1.3.2.3 Particle Firing/Sintering to Microspheres

After the pregranulation step, the free-flowing particles must then be fired/sintered to the desired density and porosity. In the experimental work performed in this study, four means of accomplishing this processing step were examined.

a. Bulk Sintering In An Inert (Nitrogen) Atmosphere

In this method the pregranulated, sized materials were placed in nickel crucibles and baked at high temperatures (300-700°C) under a nitrogen atmosphere for varying periods of time. This method of sintering the lithium oxide granulates was unsuccessfully due primarily to the amount of impurities present in the pregranulates. As is indicated in Figures 1A and 1B, small amounts of lithium carbonate (and also lithium hydroxide) in the pregranulated materials greatly lower the melting point, and thus the sintering point, of the bulk material. It was found with pregranulates formed by fluidized bed agglomeration, for instance, that the material in the crucibles melted/sintered to a solid mass at less than 450°C. Such a material is clearly unacceptable for a use (CTR reactor) where it must maintain a free flowing quality at >650°C. Pregranulates formed by pressing pellets of lithium oxide and re-crushing these pellets did resist bulk sintering up to 600°C, but yielded a poorly free-flowing product. For these reasons, furnace sintering of lithium oxide pregranulates was not used to produce microsphere samples. This method could possibly be used, however, if a properly pure lithium oxide pregranulate could be formed. Furnace firing was also used to decompose the lithium peroxide precipitate material but yielded a completely non-free-flowing Li_2O powder.

b. Propane Flame Firing of Pregranulated Material

When pregranulated lithium oxide material was fed (poured) into a propane flame, extremely free-flowing, highly spherical particles were generated. However, this material was found to be highly contaminated with lithium hydroxide and lithium carbonate. Indications are that at the attainable flame temperatures, the lithium oxide reacts with the H_2O and CO_2 flame combustion products to form these constituents. The microspheres generated resemble melted particles which spheroidize via a surface tension mechanism and solidify, rather than sintered agglomerates.

c. Hydrogen-Oxygen Flame Firing of Pregranulated Material

The pregranulated materials were fed into a hydrogen-oxygen combustion flame in the same manner as the propane flame mentioned above, in the hope that the much higher temperature of this flame would retard the reaction of the lithium oxide with the H_2O flame combustion product. This did not prove to be substantially the case. Again, an extremely free-flowing, microspherical particle was produced, but was highly contaminated with lithium

hydroxide. The lithium peroxide precipitate material was also fired by this method. It was thought that the flame would first decompose the lithium peroxide to lithium oxide and then spheroidize the oxide particle. Again, however, extensive lithium hydroxide was formed in the particle.

In both the propane and the hydrogen-oxygen flame firing of the pregranulated materials, an unacceptable microsphere product was formed. These methods did, however, suggest a method for spheroidizing lithium oxide pregranulates which hold promise for success but which could only be peripherally examined under the present research project, plasma spheroidation (below). For this reason, microspheres of the various pregranulates were generated for further study by this method as an analog to plasma spheroidation.

d. Plasma Spheroidation of Fluidized Bed Li_2O Pregranulates

A small sample of lithium oxide pregranulate prepared by the fluidized bed technique was sent to TAFA Metallization Inc.* for processing in an oxygen RF induction plasma. A small fraction of this material was processed and returned for further analysis.

1.3.2.4 Based on the foregoing examination of basic candidate processes for the fabrication of lithium oxide microspheres, integrated methods for the production of samples of microspheres of lithium oxide/hydroxide/carbonate were devised and carried out. Figure 1-3 presents a rough flow chart for these methods and the following section will detail the methods and basic physical and chemical analyses of the generated microsphere samples.

1.4 Experimental Production of Microspheres

1.4.1 Of the processes evaluated in the previous pages for the steps in the production of lithium oxide microspheres, those considered for extended experimental evaluation were organized into essentially three processing schemes, shown in Figure 1-3.

The first scheme involves the use of as-received lithium oxide powder, -325 mesh and ~95% pure, pregranulating this powder to a free flowing agglomerate in a fluidized bed, and firing this material in a propane/air or hydrogen/oxygen flame to microspheres.

The second scheme is identical to this with the exception that the pregranulation step is accomplished by first pressing the as-received powder into pellets, then crushing these pellets through sieves.

* TAFA Metallization, Inc., Bow, New Hampshire.

FIGURE 1-3 LITHIUM OXIDE MICROSPHERE FABRICATION SCHEME

The third scheme involves using reagent grade lithium hydroxide monohydrate powder as starting material, precipitating lithium peroxide by hydrogen peroxide addition, drying the precipitate to a free flowing condition, and firing this material directly in a hydrogen/oxygen flame for decomposition and spheroidation.

Both these methods produced very free flowing microsphere particles, but none produced a microsphere of sufficient lithium oxide purity, primarily due to the reaction of the lithium oxide with the combustion products of the propane and hydrogen flames.

1.4.2 One of the starting materials selected for the production of lithium oxide microspheres was, as mentioned, -325 mesh lithium oxide powder purchased from a chemical specialty retail supplier. Chemical analysis of this material, consisting primarily of titration analysis occasionally augmented by atomic absorption, showed this material to be approximately 95% pure lithium oxide, with the impurities consisting primarily of lithium hydroxide, lithium carbonate, and some magnetite. This magnetite is thought to originate from processing equipment used by the supplier.

Figure 1-4 displays a photomicrograph of this powder. The divisions shown indicate 100 microns. The powder can be seen to be a very fine crystalline material, subject to loose clumping. The powder is totally non-free flowing and tends strongly to stick to surfaces it contacts. The extreme fines readily disperse into the air upon opening a container of this material, and this effect, associated with the caustic toxicity of the lithium oxide itself towards nasal and respiratory systems, created extreme difficulty throughout the experimental program in the handling of this material. This noxious breathing effect of the powder was so pronounced in many cases that it became reason enough itself for the termination of some experimental processes (i.e., high intensity mixing para. 1.3.2.2.b). Additionally, extended skin contact with the material causes skin irritation, cracking, and rash formation. This problem was easily avoided by using plastic gloves while handling the material, but the breathing hazards were completely eliminated during experimental work only when the particular action could be accomplished inside a ventilated hood, which was not always feasible.

Due to the extreme facility with which lithium oxide captures water vapor and carbon dioxide from ambient air, care had to be taken to seal the material while not in use. This generally consisted of keeping the lithium

FIGURE 1-4 -325 MESH LITHIUM OXIDE POWDER

oxide inside a plastic bag which was in turn sealed inside a metal container. Some lithium oxide was also kept inside a standard laboratory glass bottle. These methods proved only marginally adequate since analysis of some of the oxide after about five months of such storage showed a decrease of lithium oxide content to approximately eighty-five percent.

1.4.3 The second starting material used in this study was purified grade lithium hydroxide monohydrate. This material was converted to lithium peroxide according to the procedure elaborated in Appendix A. Three variations of this procedure were conducted to generate samples in this study:

- lithium peroxide generated exactly according to the procedure in Appendix A
- lithium peroxide generated using a 45-minute stir time in step 4 of Appendix A instead of the prescribed 20 minutes. This longer stir time was to have generated larger agglomerated precipitate particles.
- lithium peroxide generated by this same procedure at the University of Wisconsin.

There were noticeable differences between the lithium peroxide samples generated at the University of Wisconsin (U.W.) and at B&W, despite both ostensibly being produced via the same procedure. The U.W. lithium peroxide was a deeper cream color than either of the two B&W samples. Additionally, and more importantly, the size distribution of precipitate particles was much more favorable in the U.W. Samples. Figures 1-5a, 1-5b, and 1-5c show photomicrographs of these three samples which demonstrate this difference. The U.W. sample shows a lower fines content and a seemingly more uniform size distribution. It can be seen from the two B&W samples that the longer stir time creates some larger particle agglomerates but does not eliminate the very fine particles. The differences in the size and size distributions shown in these photomicrographs demonstrate the sensitivity of these parameters to slight changes in the precipitation procedure.

FIGURE 1-5a LITHIUM PEROXIDE PRECIPITATED BY
THE PROCEDURE GIVEN IN APPENDIX A

FIGURE 1-5b LITHIUM OXIDE PRECIPITATED
AFTER EXTENDED STIRRING

FIGURE 1-5c LITHIUM PEROXIDE PRECIPITATE GENERATED
AT THE UNIVERSITY OF WISCONSIN

1.4.4 As referred to earlier, the purchased lithium oxide powder was experimentally pregranulated to size and free flow requirements by two methods; fluidized bed agglomeration and dry pellet pressing/sieve crushing.

1.4.4.1 In the fluidized bed agglomeration, accomplished at the testing laboratory of Glatt Air Techniques, approximately 1 Kg batches of the lithium oxide powder, the smallest possible quantity for the available equipment, were fluidized by partially dried air as droplets of liquid binder material were sprayed on the bed. The powder particles are agglomerated by the combined action of the binder and the mixing action in the fluidized bed. Through control of the rate of gas fluidization, binder spray rate, total binder added, and unit temperature, the size distribution of the agglomerated particles may be controlled.

It was found during experimentation with the fluidized bed that approximately 2% by weight of binder material to lithium oxide was sufficient to agglomerate the -325 mesh powder to the desired particle size range (100 to 200 microns). The binder material which were used were ethyl cellulose, KLUCEL-L*, and ethyl silicate. As far as was noticeable in this investigation, each binder material produced about the same binding effect on the lithium oxide powder. The use of ethyl silicate as binder material was attempted in order to retard the formation of lithium carbonate in the fired microsphere product and possibly form a lithium silicate coating on the microsphere which would inhibit microsphere bed sintering. The high melting point of lithium silicate (1204°C) would make it more tolerable than lithium carbonate or lithium hydroxide as a microsphere impurity. It was found, however, that a primary source of carbonate and hydroxide impurities occurred in the subsequent firing step and thus the use of the silicate binder in this step effected little overall improvement.

Figure 1-6 shows a photomicrograph of a typical fluidized bed granulation of lithium oxide powder, in this case using 2% by weight ethyl cellulose as binder material. The particles can be seen to resemble clumps of crystals stuck together. Note also that some of the fine Li_2O particles which cause toxicity problems for personnel working around this material still exist for this material.

*Trademark Hercules Chemical Corporation

FIGURE 1-6 FLUIDIZED BED PREGRANULATED LITHIUM OXIDE
POWDER: 2% ETHYL CELLULOSE BINDER

The agglomerates themselves at this stage are fairly easily broken up (i.e., by agitation in a kitchen-type blender), thus requiring the subsequent firing steps from a particle strength standpoint as well as considerations of sphericity, density, and porosity. The agglomerated powder does, however, exhibit a good free flow property.

As was noted previously, however, the major problem associated with the fluidized bed pregranulation was the inability, with the equipment available, to remove all water vapor and carbon dioxide from the fluidizing air of the processing unit. The lithium oxide readily reacted with these substances, forming extensive lithium hydroxide and lithium carbonate impurities which severely compromise the desired properties of the microsphere final product.

Another problem associated with this method was the loss of the finest lithium oxide particles through the bellows-type fine filter at the gas outlet of the unit. These fines could be observed visually through a plexiglas observation port to be passing through the fabric of the bellows filters.

The general problem of handling the lithium oxide powder in an open atmosphere was also experienced in this method. The transfer of the powder to and from the processing unit permitted some of the Li_2O fines to get into the ambient air causing extreme breathing irritation at times. Any commercial or laboratory facility for the processing of any significant amount of this material should provide completely enclosed, well vented, equipment for handling and transfer of the material. This problem was significant during this phase of experimentation, although not at all limited to this phase.

The type and amount of binder material to be used in pregranulating Li_2O powder by the fluidized bed technique is still another unsolved problem in this process. It is suspected, but by no means proven, that the cellulose type binders used in this experiment contributed to the formation of lithium carbonate in the pregranulate by the mechanism of an ester-base reaction between the cellulose groups and the (basic) Li_2O , decomposing the binder and forming Li_2CO_3 and LiOH . This mechanism is a possible explanation for a self-heating effect the agglomerated powders demonstrated after removal from the fluidized bed. All samples generated by the fluidized bed technique experienced a delayed temperature excursion of up to 350°F after being collected after processing. This could have been due to the decomposition of

of the binder material and subsequent exothermic reaction of the lithium oxide with the decomposition products. Since we were limited to those binder materials soluble in acetone, to enable the spray nozzle to disperse the binder over the bed, our choice of binders was limited to the three materials mentioned. Other binder materials may exist which meet processing qualifications and do not react with the lithium oxide.

1.4.4.2 In the pellet pressing/sieve crushing method for producing a sized, free flowing lithium oxide pregranulate, the -325 mesh lithium oxide powder was pressed into pellets at various pressures and subsequently crushed through a No. 70 (210 micron opening) sieve tray. It was found that for the powder used in this study the optimum press strength was approximately 4500 Mt. At pressures significantly higher or lower than this value the resulting crushed pregranulate was insufficiently free flowing and had an excessive fines content. Even when pressed at this strength, the free flowing character after sieve crushing was barely adequate and the fines content somewhat excessive.

In another sample, a dry binder material, NOPCOWAX DSM, was blended with the lithium oxide powder prior to pellet pressing. It was thought that the resultant crushed pellet might yield superior properties if free flow and fines content to the pregranulate generated without binder addition, but any improvement shown was insignificant.

Pre-sintering of the pressed Li_2O pellets in a nitrogen atmosphere furnace at 500°C also yielded insignificant improvement in pregranulate properties.

Figure 1-7 presents a photomicrograph of the pregranulate material resulting from pressing Li_2O powder at 4500 with no binder added and crushing the pellets through a number 70 sieve. This material proved adequate for subsequent flame injection but did not free flow as well as the fluidized bed pregranulate and contained more fines.

The problem of Li_2O toxicity and handling was less of a problem in this phase of work since the pelletizing and crushing operations could be carried out inside a well ventilated hood. Also, there was considerably less formation of lithium hydroxide and lithium carbonate due to lessened particle/air contact.

1.4.5 The firing of the free flowing, sized, lithium oxide/lithium peroxide pregranulates was accomplished using standard laboratory burners and a simple construction of staggered microsphere collection pans. The firing/collection equipment was constructed inside a well ventilated hood to alleviate powder toxicity problems.

FIGURE 1-7 LITHIUM OXIDE PREGRANULATE FORMED
BY PELLET PRESSING/SIEVE CRUSHING

1.4.5.1 The pregranulated powders are poured by hand through propane/air and hydrogen/oxygen combustion flames, wherein each particle is separately sintered and propelled by the flame into the staggered collector pans. In general, the smaller particles would tend to spheroidize earlier and be propelled more easily by the flame, therefore collecting in the pans more distant from the flame, whereas the larger particles or chunks tend to fall more vertically through the flame. In this manner a rough separation between the fired and incompletely fired particles is effected.

As previously mentioned, the primary difficulty associated with the flame firing technique was the reaction of a portion of the lithium oxide with the components of the combustion flame to form lithium hydroxide and lithium carbonate. Since the melting points of these materials, particularly the hydroxide at 462°C , are significantly lower than that of lithium oxide, and the phase relationships seem to indicate a rapidly decreasing mixture melting point with small amounts of carbonate impurity, their formation is clearly undesirable. It may be the formation of these materials which cause the particles to partially melt, however, and form the spherical particles shown in Figures 1-8a and 1-8b.

Figure 1-8a shows microspheres generated by the propane/air flame firing of fluidized bed pregranulated lithium oxide with 5% KLUCEL binder. The pregranulated material had been sieved prior to firing, resulting in the fairly uniformly sized microspheres shown in the photomicrograph, and the comparative lack of fines and unfired particles. The line spacing shown in the photomicrograph corresponds to $100\text{ }\mu\text{m}$.

Figure 1-8b shows microspheres generated by the hydrogen-oxygen flame firing of fluidized bed pregranulated lithium oxide with 2% ethyl silicate binder. This material was not sieved prior to firing. Titration analysis of the pregranulated material prior to firing showed 63% Li_2O , 28% LiOH , and 9% Li_2CO_3 by weight. The same analysis after firing to microspheres showed 45% Li_2O , 48% LiOH , and 7% Li_2CO_3 , clearly showing the reaction of the lithium oxide with the H_2O combustion product in the flame.

Figures 1-9a and 1-9b show microspheres generated by the hydrogen-oxygen flame firing of lithium oxide pregranulated by the pellet pressing/sieve crushing technique. Analysis of the pregranulated material showed the composition to be approximately 84% Li_2O , 14% LiOH , and 2% Li_2CO_3 . Analysis

FIGURE 1-8a MICROSPHERES RESULTING FROM PROPANE FIRING
OF FLUIDIZED BED PREGRANULATED LITHIUM OXIDE

FIGURE 1-8b MICROSPHERES RESULTING FROM HYDROGEN/OXYGEN FLAME FIRING
 OF FLUIDIZED BED PREGRANULATED LITHIUM OXIDE

FIGURE 1-9a MICROSPHERES GENERATED BY HYDROGEN/OXYGEN FLAME FIRING
OF PELLET PRESSED/CRUSHED LITHIUM OXIDE PREGRANULATES

FIGURE 1-9b MICROSPHERES GENERATED BY HYDROGEN/OXYGEN
FLAME FIRING OF PELLET PRESSED/SIEVE CRUSHED
PREGRANULATES WITH 2 PERCENT NOPCOWAX BINDER

of the microspheres after firing shows 45% Li_2O , 47% LiOH , 8% Li_2CO_3 , demonstrating again a reaction of the lithium oxide with the flame components. The photomicrographs of the material also show a fired particle much more ragged and less spherical than the microspheres generated from the fluidized bed granulated material. This effect could be due to the lower level of LiOH in the pregranulate material and thus possibly lower initial degree of particle melting in the flame. The particles are very free flowing nevertheless. The microspheres generated from the pellets pressed with 2% NOPCOWAX binder show a slightly higher lithium carbonate impurity level, possibly due to the combustion of the binder material in the excess oxygen in the flame, and subsequent generation of CO_2 which reacts with the lithium oxide.

1.4.5.2 The firing of the dried, free flowing, lithium peroxide precipitate is designed to accomplish two reactions; the thermal decomposition of the lithium peroxide (Li_2O_2) to lithium oxide followed by the sintering/spheroidation of the oxide to microspheres. It is also considered that, due to the decomposition release of gaseous oxygen, microspheres generated by this method might be more porous than those generated by the previously considered techniques. A difficulty inherent in this method is the wide size range of Li_2O_2 particles from the precipitation step, and the difficulty of controlling the particle sizes by precipitation parameters.

Figure 1-10 shows a photomicrograph of microspheres generated by this method. These microspheres show excellent free flow qualities, but a level of lithium hydroxide impurity of about 60%, higher than any other encountered method.

1.4.6 A small sample of microspheres was generated from a fluidized bed pregranulated material using an argon-oxygen plasma torch as a firing mechanism. Chemical analysis of the sample before and after firing revealed that the composition remained nearly the same. The resulting microspheres generated were not pure lithium oxide, but this was due to the fact that the pregranulate material was not pure. Figure 1-11 shows a photomicrograph of these microspheres. This result would seem to indicate that pure lithium oxide microspheres could be generated by this firing method if a suitably pure pregranulate material could be used as feed to the RF plasma torch unit. This method is being conceptually evaluated for future research into producing lithium oxide microspheres. The small volume of the sample generated thus far precludes extensive chemical or physical analysis of such a product.

FIGURE 1-10 MICROSpheres GENERATED BY THE HYDROGEN/OXYGEN
FLAME FIRING OF LITHIUM PEROXIDE PRECIPITATE

FIGURE 1-11 PLASMA FIRED Li_2O MICROSPHERES FROM
FLUIDIZED BED PRE-GRANULATED MATERIAL

1.4.7 Table 1-2 shows a summary of the results of the various microsphere fabrication methods attempted in this study. No microspheres of a sufficient purity for the intended CTF power reactor application were generated in this study, due primarily to the high reactivity of lithium oxide and its tendency to easily form lithium hydroxide and lithium carbonate with, respectively, water and carbon dioxide. Based on the foregoing results, it is surmised that the best method for the production of pure microspheres would necessarily involve RF plasma firing of the sized pregranulate since only this firing method seems not to involve reaction of the lithium oxide with flame components.

TABLE 1-2: SUMMARY OF LITHIUM OXIDE MICROSPHERE FABRICATION RESULTS

STARTING MATERIAL	PREGRANULATION METHOD	PREGRANULATE ANALYSIS	SINTERING/FIRING METHOD	MICROSPHERE ANALYSIS	COMMENTS
1. Li_2O powder, stock, alfa chemicals, -325 mesh, ~96% pure	Dry air fluidized bed granulation	<ul style="list-style-type: none"> - Ethylcellulose binder, 2.5% in acetone, total 7% ethylcellulose by weight in Li_2O - Size distribution, mesh (d = Diam.) <ul style="list-style-type: none"> d > 100 50% 100 > d > 200 40% 200 > d > 325 10% 325 > d Trace - Li_2O 64% by weight LiOH } 36% by weight Li_2CO_3 	Propane-air combustion flame	<ul style="list-style-type: none"> - 31.5% by weight Li - Size distribution <ul style="list-style-type: none"> d > 65 20% 65 > d > 100 50% 100 > d > 200 25% 200 > d 5% 	Chemical titration analysis reveals presence of lithium carbonate (Li_2CO_3) and lithium hydroxide in microsphere product as result of reaction with flame combustion products. Some ether/strong base reaction between binder material and lithium oxide is also thought to occur, producing Li_2CO_3 . Microspheres formed are very spherical and free flowing.
2. Same as above	Same as above	<ul style="list-style-type: none"> - Klucel-L 3.7% in acetone, total 5% by weight with Li_2O - Size distribution, mesh <ul style="list-style-type: none"> d > 100 10% 100 > d > 200 40% 200 > d > 325 40% 325 > d 10% - Li_2O 56% by weight LiOH } 44% by weight Li_2CO_3 	Propane-air combustion flame	<ul style="list-style-type: none"> - Size distribution, mesh <ul style="list-style-type: none"> d > 100 56% by wt 100 > d > 200 42% " 200 > d > 325 1% " 325 > d 1% " 	Particles formed are very spherical and free flowing. This sample and the one above were found to bulk sinter in a nitrogen atmosphere at around 450°C.

TABLE 1-2 (Cont'd)

STARTING MATERIAL	PREGRANULATION METHOD	PREGRANULATE ANALYSIS	SINTERING/FIRING METHOD	MICROSPHERE ANALYSIS	COMMENTS
3. Same as above	Same as above	- Ethylcellulose 2.5% in acetone, total 2% by weight to Li_2O - Li_2O 62% LiOH 34% Li_2CO_3 4%	Propane-air combustion flame	- Li_2O 50% LiOH 28% Li_2CO_3 22% - Size distribution, mesh $d > 100$ 81% $100 > d > 200$ 19% $200 > d > 325$ Trace $325 > d$ Trace - Porosity: 7.8% open ($>.044\mu$) 11.2% closed Bed density .98g/cc	Increased conversion of Li_2O , LiOH to Li_2CO_3
4. Same as above	Same as above	Same as above	Hydrogen-oxygen combustion flame	- Li_2O 20% LiOH 61% Li_2CO_3 19% - Size distribution, mesh $d > 100$ 19% $100 > d > 200$ 52% $200 > d > 325$ 6% $325 > d$ 23%	Hydrogen-oxygen flame firing does not provide high enough temperature to retard the $\text{Li}_2\text{O} + \text{H}_2\text{O} \rightarrow \text{LiOH}$ reaction. Obvious that H_2O combustion product reacts with Li_2O particles. Extremely free flowing, spherical microspheres are formed, however.
5. Same as above	Same as above	- Ethylsilicate 3% in acetone, total 2% to Li_2O - Li_2O 63% LiOH 28% Li_2CO_3 9%	Propane-air combustion flame	- Li_2O 42% LiOH 36% Li_2CO_3 22% - Porosity: 6.1% open ($>.044\mu$) 10.9% closed Bed density .98g/cc - Li_2O 45% LiOH 48% Li_2CO_3 7%	Use of silicate rather than cellulose binder did not appreciably lessen lithium carbonate formation, indicating reaction with flame combustion products is primary source of Li_2CO_3 . Same true for LiOH formation in hydrogen-oxygen flame. Again, extremely free-flowing, spherical microparticles were formed.

TABLE 1-2 (Cont'd)

STARTING MATERIAL	PREGRANULATION METHOD	PREGRANULATE ANALYSIS		SINTERING/FIRING METHOD	MICROSPHERE ANALYSIS		COMMENTS
6. Same as above	Same as above	Same as above		Hydrogen-oxygen combustion flame	- Porosity: 4.9% open(>.044μ) 11.4% closed Bed density 1.03g/cc		
7. Same as above	Same as above	- Li ₂ O	55%	Oxygen RF plasma fired	- Li ₂ O	55%	Plasma firing does not seem to decompose LiOH, Li ₂ CO ₃ , but product composition remains essentially the same as the pregranulate.
		- LiOH	39%		- LiOH	41%	
		- Li ₂ CO ₃	6%		- Li ₂ CO ₃	4%	
8. Same as above	Pellet pressing/ crushing	- No binder		Hydrogen-oxygen combustion flame	- Li ₂ O	45%	Pellets of Li ₂ O are pressed from -325 mesh powder and crushed/ sieved through a 70 mesh screen to produce a moderately free flowing pregranulate for injection through hydrogen-oxygen flame.
		- 100% through 70 mesh			- LiOH	47%	
		- Li ₂ O	84%		- Li ₂ CO ₃	8%	
		- LiOH	14%		- Size distribution, mesh		
		- Li ₂ CO ₃	2%		d > 100	66%	
					100 > d > 200	25%	
					200 > d > 325	2%	
					325 > d	7%	
					- Porosity		
					9.3% open(>.044μ)		
					17.2% closed		
					Bed density .87g/cc		
9. Same as above	Same as above	- 2% dry blended NOPCOWAX PM as binder		Hydrogen-oxygen combustion flame	- Li ₂ O	43%	
		- 100% through 70 mesh			- LiOH	44%	
					- Li ₂ CO ₃	13%	
					- Size distribution, mesh		
					d > 100	62%	
					100 > d > 200	26%	
					200 > d > 325	2%	
					325 > d	10%	
					- Porosity:		
					11.7% open(>.044μ)		
					18.3% closed		
					Bed density .76g/cc		

TABLE 1-2 (Cont'd)

STARTING MATERIAL.	PREGRANULATION METHOD	PREGRANULATE ANALYSIS	SINTERING/FIRING METHOD	MICROSPHERE ANALYSIS		COMMENTS
10. Lithium hydroxide monohydrate reagent	Precipitation of lithium peroxide - slow millipore filtration	- No binder	Hydrogen-oxygen combustion flame	- Li ₂ O LiOH Li ₂ CO ₃	29% 53% 18%	Carbonate level in microspheres is high, unknown source. Microspheres very free flowing.
				- Porosity: 6.0% open(>.044μ) 20.9% closed Bed density .76g/cc		
11. Same as above	Lithium peroxide precipitation - Buchner fast filtration	- No binder - 91.5% Li ₂ O ₂	Hydrogen-oxygen combustion flame	- Li ₂ O LiOH Li ₂ CO ₃	34% 61% 5%	Pregranulate somewhat sticky
12. Same as above	Lithium peroxide precipitation - Buchner filtration and slow heat to dryness	- No binder	Hydrogen-oxygen combustion flame			Pregranulate very free flowing

1.5 Microsphere Porosity

1.5.1 As has been mentioned previously, microspheres of the necessary lithium oxide purity were not produced in this study. Since the porosity obtained in the firing of the pregranulates depends in quantity and nature on the composition of the fired material, porosity studies of the microspheres obtained would have severely limited application to the prediction of the porous properties of a pure lithium oxide microsphere generated via the same methods, and thus detailed pore morphology analyses of the impure microspheres generated in this study were not undertaken. However, a rough idea of the kinds of porosity which might be generated in a pure lithium oxide microsphere might be useful, and towards that end, some porosimeter studies were conducted on selected microsphere samples. These results, too, are included in Table 1-2.

1.5.2 Porosity data for the selected microsphere samples were generated using a simple mercury porosimeter apparatus. This apparatus was capable of generating mercury head pressures on the order of 5000 psi, corresponding to filled open pores as low as .044 microns. The apparatus did not have any capability for the measurement of closed porosity, but an estimate of closed porosity could be roughly inferred from the porosimeter data in conjunction with settled bed density data.

Figure 1-12 shows a typical porosimeter curve of mercury head pressure versus void volume, in this case for microspheres generated by the propane firing of a pressed/crushed pregranulate with 2 percent NOPCOWAX binder. Since the particles analyzed by this method are microspheres whose pore dimensions are considerably smaller than the interstitial dimensions, a rough determination of point A in Figure 1-12, that point at which large mercury pressures are required to squeeze only small fractions of mercury into voids, should give the volume of interstitial voids only, and point B would give the determination of voids down to .044 microns. However, point B could be slightly compromised by the possibility that at these very high (~5,000 psi) mercury pressures, some crushing of the microspheres may be occurring. Thus, the values obtained for "open" and "closed" porosity given in Table 1-2 should be regarded only as approximate.

The porosity data presented in Table 1-2 seems to indicate that microspheres generated by the pellet pressing and peroxide precipitation techniques tend to be more porous, particularly for closed porosity, than those microspheres generated through fluidized bed granulation. This seems to make sense for the

peroxide since the firing step decomposes the Li_2O_2 to Li_2O and oxygen gas, which could conceivably become trapped as small closed bubbles in the microspheres, as well as spheroidizes. No explanation is offered for the difference in porosity characteristics for the other two methods.

1.6 Cost Estimate for Microsphere Production

For the purpose of obtaining a very rough cost estimate for the production of lithium oxide microspheres, the following assumptions were made:

- a. The fabrication process is assumed to consist of the steps: purchase of -325 mesh lithium oxide powder, fluidized bed granulation of the powder, and argon-oxygen plasma torch firing of the pregranulate to microspheres.
- b. The cost of -325 mesh, 98 percent pure lithium oxide powder to be \$25/lb. This figure is a compromise between the present cost, \$115/lb, due primarily to low demand, and indications that a suitably pure Li_2O powder can be generated by the peroxide precipitation method.
- c. Recycle and loss rates are comparable with those experienced in the experimental program thus far completed.

Using these assumptions, and reasonable estimates of required manhours, power charges, and overhead, a rough estimate for a small scale (~50,000 - 100,000 lb/yr) production of microspheres would be \$30-\$35/lb. It must be emphasized here that the processing costs are themselves very approximate. It is also obvious that the major cost is that of the lithium oxide raw material. If, for instance, the peroxide precipitation method could be adjusted to yield a well-sized precipitate in the desired size range for direct plasma firing, the resultant ability to use lithium hydroxide monohydrate as starting material would dramatically reduce the raw material cost. The ability to generate a reasonably accurate estimate of the cost of producing commercial quantities of the microspheres must await further definition of optimum microsphere fabrication process parameters.

1.7 Summary and Conclusions

1.7.1 The fabrication of lithium oxide microspheres is severely complicated by the high chemical reactivity of lithium oxide, especially with water and carbon dioxide, forming, respectively, lithium hydroxide and lithium carbonate. The presence of significant quantities of these materials in the finished microsphere product is undesirable because of the lowered sintering and melting points they would induce in the microsphere bed. Any method used for the fabrication of lithium oxide microspheres must be chosen, therefore, to minimize exposure of the lithium oxide to water or carbon dioxide.

FIGURE 1-12 TYPICAL POROSIMETER CURVE FOR MICROSPHERE SAMPLES

None of the fabrication methods attempted in this study produced adequately pure lithium oxide microspheres, but the research did indicate promising approaches for work. The essential two steps for lithium oxide microsphere fabrication are pregranulation or sizing of lithium oxide (or peroxide) to the approximate desired microsphere size and the firing of these pregranulates to hardened microspheres. Three methods of "pregranulation" were judged adequate in this study: fluidized bed pregranulation, pellet pressing/crushing, and lithium peroxide precipitation. Of these, the best in terms of pregranulate particle size, size distribution, and free flowing quality is the fluidized bed method. However, a capability to fluidize with a completely water vapor and carbon dioxide free fluidizing gas is necessary for its feasibility. The equipment used in this study did not have that capability.

The firing of the pregranulated material to hardened microspheres was done in this study using primarily combustion flames. This proved inadequate due to the reaction of the lithium oxide with the flame combustion products; however, it did indicate the feasibility of the mechanics of microsphere formation in the flame. There is thus a strong indication that the pregranulated materials can be fired to a suitably pure lithium oxide microsphere using a radio-frequency induced oxygen or oxygen-argon plasma torch.

1.7.2 At times during this research, extreme difficulty was encountered in the handling of the lithium oxide, hydroxide, and peroxide powders due to their noxious effect on skin surfaces and respiratory systems. This was especially true of the -325 mesh lithium oxide starting material obtained commercially. The ability of this material upon exposure to disperse in ambient air caused considerable difficulty when handling of the material outside a hood was necessary. A lesser problem than the breathing difficulties, but still significant, was that of extended skin exposure to a fine Li_2O powder causing rashes and skin cracking.

1.7.2 The porosity and pore morphology of the microspheres created by the type of processes considered in this research depends a great deal on such things as the nature, shape, and composition of the pregranulated material, and the temperature, residence time, and type of flame for the firing step. In particular, since the Li_2O composition of the materials in this study did not indicate a sufficient purity, the porosity data which were obtained would be impossible to accurately extrapolate to a pure lithium oxide microsphere, this

also being the primary reason why extensive pore morphology studies were not conducted on the impure microspheres which were generated.

1.7.3 The studies reported herein have not by any means exhausted the possible methods for the creation of pure lithium oxide microspheres. In particular, organic sol-formation and -extraction and, possibly, flame spraying from an Li_2O melt, to mention just two, may have possibilities as microsphere fabrication methods but were not investigated in this study.

1.8 Recommendations

1.8.1 It is recommended that the work on lithium oxide microsphere fabrication be continued along the lines indicated by the results of this study. Specifically, studies should continue into characterizing the pregranulation methods of fluidized bed agglomeration, pellet pressing/crushing, and lithium peroxide precipitation. Studies in the firing of the pregranulates to microspheres should continue using an oxygen or argon-oxygen plasma for microspheroidation.

1.8.2 Further studies should be initiated regarding the basic physical chemistry of lithium oxide. In particular, a study of the three-component phase equilibrium of the lithium oxide-lithium hydroxide-lithium carbonate system would be extremely valuable in evaluating the tolerable impurity limits in the microspheres and characterizing microsphere sintering behavior both in microsphere fabrication systems and in the CTF reactor blanket environment. Such a phase-equilibrium study could be done using thermogravimetric (TGA) and differential thermal analysis (DTA) techniques.

1.8.3 After processing parameters have been established which permit the fabrication of suitably pure lithium oxide microspheres, studies should be made concerning the effects of parameter alterations on the porosity and pore morphology of the resulting microspheres, with the ultimate purpose of establishing an ability to produce microspheres of a specified strength, size, size distribution, and pore morphology.

REFERENCES

- (1) "Gravity Circulated Solid Blanket Design for a TOKAMAK Fusion Reactor", D. K. Sze, et al, Proc. Second Topical Meeting on the Technology of Controlled Nuclear Fusion, Richland, Washington, September 21-23 1976.
- (2) "The Preparation of Lithium Peroxide and Oxide", P. Pierron, Bull. Soc. Chin 6, 235-8 (1939).
- (3) "Preparation of Anhydrous Lithium Oxide", J. Aubrey and C. Gleitzer, Bull. Soc. Chin. France 1957, 809-10.
- (4) Lithium Oxide, A. L. Vulikh, M. I. Makovetskii, and L.O. Prikhod'ko, USSR 146,730 April 28, 1962.
- (5) "The Preparation of Anhydrous Lithium Oxide and Hydroxide from Lithium Hydroxide Monohydrate, A. L. Bulikh, L.D. Prikhod'ko and M.I. Makovetskii, Prom. Khim Reaktevov, Inform Byul. No. 2, 23 (1963)
- (6) Phone conversation with Lithcoa
- (7) "Etude du systeme binaire carbonate-oxyde de lithium"
G. Papin, M. Michaud, R. Bouaziz, C.R. Acad. Sc. Paris (12 May 1969)
Serie C - 1691.
- (8) "Equilibrium Diagram of the Lithium Carbonate - Lithium Oxide System"
M. V. Smirnov, I. Y. Lyubimtseva, L. A. Tsiovkina, Y.N. Krasnov
Russian Journal of Inorganic Chemistry 16(1) 1971
- (9) C. Kroger & E. Fingas, E. Z. Anorg. Chem. 1935, 224, 289-304.
- (10) Abou-El-Agm, J. Soc. Glass Technol. 1953, 37 168-81T.

APPENDIX A

PROCEDURE FOR THE PRODUCTION OF LITHIUM PEROXIDE

The following procedure for the production of lithium peroxide powder, from lithium hydroxide starting material, for use in further experimentation, was used in this research project:

1. A large gloved bag, with internal structural supports, is used as the experimental environment. All necessary equipment and chemical materials were placed in the bag, which is then sealed and purged for two hours with nitrogen gas. A small nitrogen purge is maintained during all work inside the bag.
2. 65 grams of lithium hydroxide monohydrate ($\text{LiOH} \cdot \text{H}_2\text{O}$, Fisher Chemical, purified) is poured into 63.5 grams of 30% hydrogen peroxide (Fisher Chemical), and stirred by hand.
3. The temperature of the mixture rises almost immediately to approximately 60°C , then falls rapidly to approximately 23°C . The temperature rise also corresponds to a noticeable thickening of the mixture which requires some effort to maintain mixing. The temperature decline corresponds to a marked thinning effect of this mixture.
4. A magnetic stirrer is then placed in the reaction-beaker and the mixture is gently stirred for approximately 20 minutes. The mixture has by this time turned a creamy, off-white color, corresponding to the lithium peroxide precipitate formation.
5. 140 ml of ACS grade methanol is then added to the mixture, stirred for 20 minutes, then filtered in a Buchner funnel with #2 filter paper, attached to an aspirator.
6. After approximately 11 minutes of filtration, the cream colored precipitate was washed with 20-30 ml of methanol. This washing was repeated after another 4 minutes of filtration. The temperature rose to 32°C and maintained there.

7. The filter cake was then kept under aspiration for about one hour to remove the bulk of the liquids.
8. The wet cake was then placed in a petrie dish on a hot plate and warmed, with occasional stirring, at 60-70° for 45 minutes, until a dry, free flowing, cream colored powder formed.

2.0 Task II - Nuclear Steam Supply System Components

2.1 Objectives of Task II: The objectives of this task are as follows:

- Develop preliminary conceptual design of steam generator
- Develop preliminary conceptual design of reheater
- Specify major components required to incorporate heat storage capability needed to provide continuous steam generation at full load
- Identify required R&D

2.2 Task II Work Scope: This work consists of the following three phases:

PHASE I - Establish Reference Groundrules and Parameters

This phase has three sub-phases. The first sub-phase will involve an initial listing of design parameters, groundrules and assumptions based on work performed on this concept prior to submittal of this proposal. In the second sub-phase this information will be reviewed by experienced Babcock & Wilcox Company people and others, as required for comments and recommendations. The third sub-phase will incorporate the resulting comments and recommendations into a final set of design parameters and groundrules. This will be the basis of work to develop reference component designs (Phase II) from design concepts begun prior to submittal of this proposal.

PHASE II - Complete Preliminary Design of Steam Generator, Reheater and Heat Storage Components

In this phase, component conceptual designs will be adjusted and completed in accordance with the groundrules and parameters developed by Phase I above. These designs will be reviewed by experienced B&W people and others as required. This work will generate information in the following areas:

- Thermal hydraulic design
- Steam cycle selection
- Materials selection
- Component containment (casing design)
- Tritium diffusion to steam
- Steam leak monitoring and isolation
- Design parameters and groundrules
- Applicability of current technology
- Inspection and maintenance considerations
- Heat storage features to allow continuous steam generation at full load between reactor "burn" periods

PHASE III - Identify Required Research and Development Work

This phase will scope the research and development work considered necessary to complete development of the component designs covered by this task.

2.3 Work Performed During September 1977

Work performed under Task II this month took place in the following areas:

- o Completion of horizontal tube compact steam generator design
- o Modification of NSS arrangement to be compatible with above steam generator design
- o Completion of Design Review and R&D comments by Alliance Research Center
- o R&D work scope related to Pump and Heat Exchanger Design for future U of W Li_2O engineering test loop

These are described in more detail in the following subsections.

2.3.1 Completion of Horizontal Tube Steam Generator Design

The horizontal tube steam generator design, briefly referred to in the August report is shown in fig. 2.3-1 of this report. This design utilizes a unique arrangement incorporating special features from LMFBR, PWR and fossil fired steam generator technology to provide a maximum degree of reliability, maintenance ease and freedom from steam leakage. Each design consideration listed under Phase II on the preceding page has been carefully considered with the exception of "heat storage features"; which do not pertain to the steam generator design. Comments on each of these categories are given below.

2.3.1.1 Thermal/Hydraulic Design

This steam generator combines good thermal/hydraulic conditions on both the steam and Li_2O sides.

On the Li_2O side (shellside) a horizontal tube array on triangular pitch will naturally produce mixing in the flowing bed to provide higher heat transfer coefficients than would be attained with Li_2O flowing past vertical finned tubes at the same velocity. Thus the achievement of an adequate heat transfer coefficient between Li_2O and steam generator tubes at lower flow velocity should produce less particle attrition and a lower possibility of tube metal wear.

In addition, flow over horizontal tubes may prove to be less sensitive to fines from the standpoint of flow blockage due to "bridging". Thus, adequate heat transfer and flow characteristics may be retained with higher levels of fines in the flowing bed of Li_2O microspheres. This tends to reduce severity of system operating constraints.

Another advantage of this arrangement is that it should tend to provide a more uniform Li_2O flow distribution than would be the case for the previous vertical finned tube design (Fig. 2.3-2 in August 1977 Monthly Report).

On the tube side, the steam generator has been designed to utilize natural circulation. The circulation ratio of 5 to 1 is considered ideal for long term dependable operation with a minimum of feedwater chemistry requirements. Avoidance of film boiling at the top of the tubes in the latter half of each tube is achieved by a sinuous tube shape as shown in Fig. 2.3-1. This is considered necessary in this portion of each tube where steam quality will vary from 10 to 20 percent (mid-point to outlet).

Elevation of the steam separating drum above the tube bundle is about 35 feet to provide more than adequate circulation. Steam separating equipment inside this separating drum is of the type used in other nuclear steam generator designs and is conservatively rated in this application.

2.3.1.2 Steam Cycle Selection

A desire to simplify steam generator design requirements and avoid need for a reheater was one factor in selecting a saturated steam cycle for this Tokamak application. This permitted lower operating temperatures and greater attention to design details aimed at improving reliability, etc. Lower operating temperatures provided added assurance that low alloy steels would be adequate for steam generator construction.

The above, together with factors related to steam turbine design, microsphere fabrication, etc. is discussed in Section 2.3.2 of the July 1977 Monthly Report.

2.3.1.3 Materials Selection

The steam generator would utilize typical steam generator materials of construction such as listed below.

Component Part	Material	ASME Designation
Steam Generator Tubes	Croloy 2 $\frac{1}{4}$	SA213 Type 22
Tube Supports and Baffles	Croloy 2 $\frac{1}{4}$	SA387 Type 22
Tube Sheets	$\frac{1}{2}$ MO Low Alloy Stl.	SA508 Class 2
Spherical Heads	$\frac{1}{2}$ MO Low Alloy Stl.	SA533 Gr. B Class 1
Riser & Downcomer Pipes	Carbon Steel	SA106 Gr. C
Steam Separator Drum	Carbon Steel	SA516 Grade 70

These carbon and low alloy steels are relatively inexpensive when compared to austenitic stainless steels or high nickel alloys. Their use in the steam generator and other components of the NSS will help avoid putting a heavy demand upon chromium and nickel resources for Tokamak power plant construction (as typified by some earlier designs not using Li_2O).

2.3.1.4 Steam Generator Casing

A cylindrical casing is used in order to simplify attachment to tube sheets and Li_2O piping. Segmental plates attached thereto provide a simple straightforward method of positioning and supporting flow guide plates on each vertical side of the tube bundle, the center tube support plate and end plates (dummy tube sheets) forming the leak monitoring spaces.

If loads imposed by connecting Li_2O ducts and steam piping are low, the casing can be lightly constructed and protected from differential expansion stresses imposed by the tube bundle through use of "omega" expansion seals as shown on Fig. 2.3-1. On the other hand, if piping and duct loads are high, a stronger casing will be required. In this event, the omega seals would be eliminated since differential expansion stresses and piping loads could then be controlled by longitudinal tube flexibility imparted by the sinuous tube shape previously mentioned without danger of overstressing the casing or the steam generator tubes.

2.3.1.5 Tube Integrity

When liquid metals are used in Tokamak NSS intermediate loops, tube integrity involves avoidance of tritium diffusion and prevention of steam to sodium leaks.

In the Li_2O case, diffusion of tritium thru tube walls should not be a serious problem since most of the tritium inventory will be contained in the microspheres and in molecular bond with lithium or oxygen contained therein. Tritium present in the blanket helium should preferentially react chemically with the microspheres or diffuse into the microsphere surface due to the chemical kinetics of lithium hydroxide formation and an extremely large microsphere surface area in comparison with steam generator tube surface area. Thus one important factor requiring use of an intermediate cooling loop is avoided.

Regarding steam leaks to the moving bed, it is noted that the possibility of a dangerous explosion (as with liquid sodium) is avoided since hydrogen is not evolved in the ensuing chemical reaction. Thus, an intermediate loop is not needed to prevent transfer of pressure pulses to the reactor caused by such an explosion.

In view of the above, it was felt that an intermediate loop could be avoided if the steam generator incorporated the following:

- o Double walled (duplex) tubing to avoid thru wall crack propagation between tube wall inspections.
- o Shell side gas filled compartments adjacent to tube sheets to facilitate early detection of leaks thru tube to tube sheet weld defects or failures and minimize amount of escaped water or steam available for contact with the flowing Li_2O microsphere bed.
- o Sinuous tube shape to impart longitudinal tube flexibility needed to avoid large loads on tube to tube sheet welds and ensure nucleate boiling throughout.
- o Short tubes with smooth inner and outer surfaces to permit rapid, effective eddy current inspection of tube walls from either end. Visual inspection of inner tube ID is also facilitated.
- o Use of natural circulation of steam and water with large steam separator drum to provide water storage needed to minimize thermal cycling of pressure parts during time intervals when reactor power is zero.
- o Differential expansion between adjacent tubes, a potential difficulty if bed flow develops non-uniformities, is relieved by built in flexibility of sinuous tube shape.
- o Use of a 5 to 1 water/steam circulation ratio to provide optimum tube wall operating environment on I.D.

2.3.1.6 Steam Leak Monitoring and Isolation

The steam generator utilizes a monitored gas filled space between end baffle (dummy tube sheet) and tube sheet to monitor leaks through tube to tube sheet welds at each end. Steam generator tubes are lightly expanded into dummy tube sheets to minimize leakage of helium through holes in this structure. Helium gas is pumped out of these spaces by oilless metal diaphragm compressors and circulated through moisture detecting equipment and dryers.

Return flow to the steam generators passes partly to the monitoring space and partly to the main Li_2O space in the proportion required to provide a small helium flow thru the baffles (dummy tube sheets) into the monitoring space. This largely avoids passage of steam into the Li_2O caused by a small leak typical of tube to tube sheet weld failure.

Walls of the duplex steam generator tubes will be placed in close proximity by co-drawing. This, plus the use of tube expanders in tube sheets and end baffles should satisfactorily avoid significant leakage through a tube to tube sheet weld defect, flow between tube walls and thence through a crack in the outer wall of the same tube. Such dual failure involving the same tube will be quite unlikely, especially since inservice eddy current inspection of the tube walls should detect a crack in the outer wall before it proceeds through the entire outer wall thickness.

The same general argument holds regarding cracks in inner and outer walls of the same tube. Such a condition should be avoided by early detection. Furthermore, cracks or pits occurring in both walls at adjoining locations is quite remote.

2.3.1.6 Design Parameters and Groundrules

To avoid confusion with previous designs considered in this study. Parameters for the final reference steam generator design (Fig. 2.3-1 this report) are tabulated below.

MWt	200
Li_2O Temp Entering - $^{\circ}\text{C}$	500
Li_2O Temp Leaving - $^{\circ}\text{C}$	350
Li_2O Flow/Generator - Lb/Hr	6,586,200
Steam Pressure Lv Drum - psia	1,100
Steam Flow Lv Drum - Lb/Hr	899,707
Feedwater Temp - $^{\circ}\text{F}$	450
Tube OD - in	.75
Combined wall thickness - in	.10
Tube I.D.	.55
Max Li_2O velocity over tubes - ft/sec	3.0
No tubes	4818
Active length - ft	7.33
Active heating surface - ft^2	6,937
Li_2O conductance - $\text{BTU}/\text{Hr } ^{\circ}\text{F ft}^2$	1,070
Tube wall cond. DTV/Hr $^{\circ}\text{F ft}^2$	2,580
Wall interface conductance - $\text{BTU}/\text{Hr } ^{\circ}\text{F ft}^2$	1,840
Nucleate Boiling Conductance - $\text{BTU}/\text{Hr } ^{\circ}\text{F ft}^2$	10,000
Overall conductance - $\text{BTU}/\text{Hr } ^{\circ}\text{F ft}^2$	508
Required heating surface - ft^2	6,306
Design Margin - ft^2	631
Riser Pipe OD - in	36

Downcomer pipe OD- in	18
Feedwater line OD - in	10
Steam line OD - in	12
Separator Drum Height - ft	17.3
Separator Drum OD - ft	8.9
No primary and secondary separators in drum	31 each

The Li_2O coefficient was computed on basis that the bed was free flowing with an equivalent viscosity of 1 centipoise using the following equation from McAdams (Eq 8) P. 229 2nd edition.

$$Nu = .33 (Pr)^{1/3} (Re)^{0.6}$$

Values of bed thermal conductivity and bed specific heat were assumed to be .448 BTU/Hr °F resp. as given in the June 19, 1977 Monthly Report.

Velocity between horizontal tube rows was set at 3.0 ft/sec. This flow was selected because it gave a computed flow resistance equal to the height of the tube bank. This tends to provide stable, uniform flow not dependent upon head produced by a mass of microspheres above the tube bank. As with the heat transfer calculations, flow friction through the tube bank assumed free flow bed characteristics and applicability of equations (18) and (19a) on page 126 of McAdams (2nd Edition) which was recommended for flow of liquids or gases across staggered tube banks. These equations are given below:

$$F = 4f'' \frac{N}{V_{\max}^2 / 2g}$$

$$f'' = \left[.23 + \frac{.11}{(x_i - 1)^{1.08}} \right] \times \left[\frac{(D_o G_{\max})}{\mu_f} \right]^{-.15} \quad \text{where}$$

F = Head loss - Ft of fluid bed

N = No of tube rows in bank over which bed flows

$V_{\max}$ = Velocity thru space between tubes - ft/sec

x_i = Ratio of tube pitch to tube dia

D_o = Tube Dia - ft

$G_{\max}$ = Bed mass velocity - Lb/Hr ft² = $\rho V_{\max} \times 3600$

ρ = Bed density (60 Lb/ft³)

μ_f = Bed viscosity - Lb/Hr ft (2.42)

Use of the above heat transfer and fluid flow expressions based upon maintenance of free flowing bed characteristics is tentatively justified by agreement with sparse heat transfer data for flowing beds reported in the June 1977 Monthly Report under Section 2.3.2.

2.3.1.7 Applicability of Current Technology

Since development of practical fusion power is generally considered by many to be the greatest undertaking yet attempted by mankind, it is absolutely essential that expenditures and time schedules be reduced as much as possible by selection of practical design approaches for various systems and components which utilize current technology to the maximum possible extent. This is of course a major advantage claimed for the Li_2O moving bed blanket concept for fusion reactors. As described in previous subsections, the steam generator design and steam cycle selection have been directed toward this goal (together with minimum use of materials resources).

2.3.1.8 Inspection and Maintenance Considerations

Easy, effective steam generator inspection and maintenance is of course essential if one is to satisfactorily avoid the possibility of steam leakage into the Li_2O moving bed. Principal features to achieve this goal are listed below:

1. Short tubes without U bends accessible for eddy current inspection from either end.
2. Duplex tube walls to prevent thru-wall crack propagation between inspections and reduce wall thermal stress.
3. Use of helium gas buffering and leak monitoring to detect and divert steam leakage due to tube to tube sheet weld failure.
4. Avoidance of "non draining" pockets in steam generator tending to retain Li_2O microspheres - thereby lowering radiation levels to be contended with during inspection and maintenance.
5. Use of sinuous tube shape allowing reduced stresses associated with differential thermal expansion or externally applied loads.
6. Avoidance of tube wall thermal stresses typical of steam generator designs absorbing heat from flowing liquid metal.

2.3.2 Modification of NSS Arrangement (Fig. 2.3-2)

Final definition of steam generator dimensions as shown on Fig. 2.3-1 in this report (B&W Dwg. No. EDO-92877F-0) required that B&W Dwg. EDO-82477B-0 be modified to indicate general proportions of connecting Li_2O ductwork and show a recommended location for the steam generator moisture separating drum. This location can of course be adjusted to make room for other reactor components such as neutral beam injection equipment, etc.

Introduction of a circulating pump in the steam generator downcomer pipe can be used to reduce pipe size below 36" OD and/or to locate the steam separating drum in a more remote location. Pumps of this type are routinely used in some designs of fossil fired steam generators and in BWRs under construction in Sweden.

They are generally described as "wet rotor" pumps. Motor windings operate in cooled pressurized water, there are no shaft seals and bearings are water lubricated. Reliability is outstanding.

2.3.3 Design Review and R&D Comments by B&W, Alliance Research Center

The appendix to this report documents correspondence received from this organization in connection with design review and R&D definition requirements of Tasks II and III of this contract.

It will be noted that comments received in August relating to steam generator design were evaluated and utilized where applicable, in the final steam generator design described herein.

2.3.4 Work Scope Related to Equipment for Future University of Wisconsin Li_2O Engineering Test Loop

The University of Wisconsin has outlined a preliminary hardware development phase involving a simplified isothermal test loop to study reactor blanket hydraulic design and obtain preliminary data related to loop operation (wear, attrition, etc.). As part of Task IV of this contract, B&W has prepared the outline of a follow-on work scope associated with provided designs of the components needed to modify this isothermal Li_2O test loop so that it can be operated at a later date with heat transfer and heat transport conditions considered typical of future fusion power reactor operation. This work would consist of the following tasks.

- I. Evaluate hydraulic characteristics of two or three candidate tube arrangements and select best one for use in loop heat exchange components.
- II. Design and estimate cost of heater and cooler to be used in modification of the U of W isothermal Li_2O test loop.
- III. Design and estimate cost of Archimedes lift pump(s) needed to circulate Li_2O in the modified loop.

These are described in further detail below.

2.3.4.1 Task I Evaluation of Tube Arrangements for Li_2O Heat Exchange

The use of horizontal tubes in staggered array will minimize the vertical distance through which the Li_2O bed must flow in a given steam generator application. Such an array will also tend to have better Li_2O flow distribution than can be expected with vertical finned tubes. In addition, the natural mixing action will tend to optimize Li_2O heat transfer.

The purpose of the proposed tests will be to determine an optimum tube spacing so that:

- a. Gravity will produce the required flow rate over each row of tubes.
- b. Li_2O flow remains in contact with entire tube periphery of each row.
- c. Tendency for dead pockets to form at top of each tube is minimized or eliminated.
- d. Adequacy of heat transfer performance is roughly established for the optimized tube arrangement.

Items a, b, and c will be determined by bench scale plastic flow models using a microsphere material adequately similar in flow characteristics to Li_2O microspheres. Item d will be roughly confirmed by an electrically heated tube array in a bench scale test apparatus.

2.3.4.2 Task II Design of Loop Heater and Cooler

Since the modified loop will not receive heat by neutron slowing down, it will be necessary to include a heater to provide the necessary thermal input. This component, although not needed to develop design and operating information for steam generators (the cooler will provide this) will furnish data for future design of heaters and regenerative heat exchangers which might possibly be required to heat and cool Li_2O sidestreams associated with tritium extraction. The

loop heater may well incorporate heating by electrical energy to obtain basic heat transfer data for heating Li_2O flowing beds.

The cooler will be designed to evaluate steam generator design features associated with the following:

- o Thermal/hydraulic performance of flowing Li_2O beds in the Tube Bank
- o Tube leak detection
- o In-service inspection of tubes
- o Tube erosion or corrosion
- o Li_2O surface fouling by ultrafine Li_2O particles
- o Workability of tube/tubesheet leak detection system
- o Tube wall temperature variation possibly caused by azimuthal variation in Li_2O heat transfer or thermal cycling caused by alternation between film and nucleate boiling.

The extent to which this information can be gained will of course depend upon loop size and the funding available to build, instrument and operate the loop. It is possible that a future larger loop will be needed to obtain more information in the above categories to provide the basic technology for reactor equipment hardware. Thus, the work contemplated in this task is intended mainly to establish designs to demonstrate concept feasibility.

2.3.4.3 Task III Archimedes Lift Pump Design and Cost Estimate

The Archimedes lift pumps required for this concept are not apparently offered by current suppliers of materials handling equipment. However, the mechanical drive components of vertical screw lift pumps should be applicable after modification to meet the special requirements of handling Li_2O at reactor operating temperatures.

If an Archimedes lift pump has already been used in the U of W isothermal test loop, the necessary information will be available to permit design of pumps to supply the extra lift required in the modified loop. If not, it will be necessary to:

- o Build and operate bench scale models of Archimedes screw designs to develop the required technology using representative microspheres.
- o Determine adoptability of screw lift pump drive components and the cost of making the required modification.

The requisite design information obtained by either of the above methods will then be used to design and price the required pumping equipment.

3.0 Task III - Particle Transport System Design

3.1 Objectives of Task III - The objectives of this task are as follows:

- Identify preliminary conceptual design of particle transport system
- Identify required research and development work.

This work will consist of the following two phases:

PHASE I - ESTABLISH REFERENCE GROUND RULES AND PARAMETERS

This phase, which parallels the work performed in Phase I of Task II, will consist of an initial listing of design parameters, groundrules, and assumptions based on work performed on the concept prior to this contract proposal. In the second subphase, this material will be reviewed by experienced Babcock & Wilcox Company people and others, as required, for comments and recommendations. The third subphase of this phase will incorporate the resulting comments and recommendations into an approved set of design parameters and groundrules, including a reference method of particle transport.

The descriptive matter generated will include information on the following subjects:

- o Applicability of current technology
- o Particle attrition
- o Pumping power
- o System component wear
- o Component design
- o Provision for continuous steam generation.

PHASE II - IDENTIFY REQUIRED RESEARCH AND DEVELOPMENT WORK

This phase will scope the research and development work considered necessary to complete development of the particle transport system and components.

3.2 Work Performed During September 1977 - Task III

Work under Task III took place in the following areas:

- o Alliance Research Center (B&W) review of Archimedes lift pump design with commentary on R&D requirements.
- o Outline pump design work required in connection with Li_2O engineering test loop planned by University of Wisconsin (Sec. 2.3).
- o Conception of a more advanced particle lift pump design utilizing adaptation of multi-stage axial pump principles in a vertical lift tube.

Work in these areas is described in the following subsections, in an appendix section of this report and in 2.3 above.

3.2.1 Alliance Research Center Comments Related to Task III

This material (see appendix) confirms selection of the Archimedes lift pump (evolved by this study) as being the type most suitable for the intended purpose. The R&D requirements that were identified are first of a kind engineering projects with the major difficulties associated with design of shaft seals and bearings.

The comments did not include recommendation that another type of lift pump be considered for this application. This tends to confirm our previous conclusion that currently available lift pump designs have one or more serious shortcomings when considered for this application.

3.2.2 Brief Description of Advanced Vertical Tube Lift Pump

A major obstacle to the use of moving microsphere beds for heat transfer and heat transport applications in closed loops has been the availability of a practical pumping method to circulate the bed. This problem is somewhat simplified if gravity is used to produce bed flow through system heat exchange components and piping. The pumping device is then required only to lift the bed flow from a lower to a higher elevation so that it can resume circulation through the system. Such lifting avoids the need for transfer of kinetic energy between particles and flowing gas streams with all the problems associated therewith.

In addition to the Archimedes type lift pump evolved by work on this contract and the mass lift method as used in certain petrochemical applications, there may be a third type of lift

pump having the characteristics of low attrition, low wear, low mechanical maintenance, etc. needed for moving bed heat transfer and heat transport applications.

If pump work is imparted to a flowing bed of microspheres by a rotating pump impeller, it is in the form of kinetic energy imparted mainly to the particles (over 99%). If bed flow is to be produced by gas pressure, one is faced with the extremely difficult problem of transferring kinetic energy from particles to gas and then converting gas velocity to pressure. However, if the velocity imparted to the particles is directed vertically upward, the kinetic energy is automatically converted to head (lift) in a relatively efficient, simple manner. On this basis one might visualize a lift pump resembling a multistage axial flow compressor with stationary and rotating blade rows in each stage. With a maximum relative speed (rubbing speed) of say 8 feet per second between blades and microspheres, each stationary blade row (diffuser) could elevate bed particles six inches as upward particle velocity dropped from eight to about six feet per second. At this elevation it would enter the next rotating blade row (above) where an additional six inches of lift would occur and absolute exit velocity was again eight feet per second. At this point, the particles would again enter a diffuser section having vanes which directed the particles in an upward direction to achieve the next six inch increment of total lift.

Funding limitations of this Contract preclude further work on this pump concept to develop a conceptual design with appropriate graphic representation (drawings). However, it is strongly urged that this pump concept be evaluated should future work be authorized.

Task IV - Interface Information for University of Wisconsin

4.1 Objectives of Task IV

This task has the purpose of supplying and interchanging information developed from this independent research effort to the University of Wisconsin as required to maintain a well coordinated joint effort in developing the Li_2O moving bed Tokamak Fusion Power Reactor concept. Information interchange will be necessary in the following areas:

- o General NSS parameters
- o NSS arrangement
- o Microsphere information
- o Steam leak monitoring and isolation
- o Inspection and maintenance

4.2 Work Performed During September 1977

On September 8 and 9, 1977, the writer, Dr. T. A. Thornton of LRC, and our chemistry consultant, Mr. E. H. Dewell, visited the University of Wisconsin.

U of W representatives involved in discussions with B&W were listed below:

Dr. Robert W. Conn - Director, Fusion Technology Program
Dr. G. L. Kulcinski, Asst. Director, Fusion Technology Program
Dr. E. Larsen, Head, Analytical Chemistry Dept. of U of W
Dr. D. K. Sze, Associate Scientist Fusion Technology Program
Dr. Said A. Kahlik - Associate Scientist Fusion Technology Program
Dr. Igor Sviatoslavsky, Associate Scientist Fusion Technology Program
J. Wrasel, Analytical Chemistry Department
M. Olvert, Graduate Student

All of the above attended a two-hour meeting on Thursday, September 8, 1977, during which

- o Discussions of chemistry problems associated with Li_2O microsphere fabrication were ably handled with U of W people by T. A. Thornton and E. H. Dewell.
- o Copies of NPGD's input to the August monthly report (Tasks II and III) were distributed and the contents briefly presented. Our work on these tasks was favorably accepted.

T. A. Thornton and E. H. Dewell, also spent considerable time in the U of W chemistry labs with Ms. Wrasel, Mr. Olvert, and Dr. Larsen. A possible simplification in the process of microsphere production was discovered as the result of observing some of the work being performed. Please refer to trip reports by Thornton and Dewell under Task I for further details.

Work in this area also included phone conversations with U of W in connection with microsphere R&D, plant parameters, future R&D requirements, etc.

5.0 Task V - Reporting

This letter report is the fifth and last monthly letter report, thereby completing the reporting requirements of this contract.

To	D. C. SCHLUDERBERG - PRODUCT DEVELOPMENT, NPGD, LYNCHBURG	
From	J. B. KITTO - STEAM GENERATOR TECHNOLOGY SECTION, ARC	RC-1 (Rev. 1)
Cust.		File No. or Ref. 5535-02
Subj.	COMMENTS ON POSSIBLE R&D REQUIREMENTS FOR THE LITHIUM OXIDE MOVING BED FUSION REACTOR NUCLEAR STEAM SYSTEM	Date SEPTEMBER 26, 1977

This letter to cover one customer and one subject only

1.0 INTRODUCTION

As part of its review on the Lithium Oxide Moving Bed Fusion Reactor Nuclear Steam System Concept being developed by NPGD for ERDA, ARC has been asked to make recommendations as to possible R&D requirements which may have to be fulfilled during the development of such a system. The most recent version of the moving bed NSS as described in Reference 1 has been reviewed for such R&D requirements, and the results of this review are briefly summarized below.

The following discussion is broken down into four major areas:

- Potential areas requiring R&D
- Studies of basic bed parameters
- Studies of system parameters
- Closure

2.0 POTENTIAL AREAS REQUIRING R&D

In the development of any new system which incorporates a totally new technology or applies an existing technology to a new application, there are certain areas which will require some form of R&D. In the case of the fusion reactor NSS concept under study, a moving bed is used as the intermediate heat transfer medium in a nuclear steam system. Such a method of transferring heat comes under the heading of a "new" unexplored technology. At the same time, the Archimedes lift pumps and horizontal steam generator represent major applications of older technologies to a new system. A brief review of this

cc: HRCarter - ARC
JHKidwell - ARC
MLukasik - CRD
TAMcNary - ARC
WJOberjohn - ARC
GBWatson - ARC *GBW*
CPWelch - ARC *CPW*

nuclear steam system reveals a number of potential problem areas which will require R&D activity. These areas are listed below. Though the list is by no means exhaustive, it does indicate the type of problem areas which can be expected during the NSS development. The order in which these areas are listed is not an indication of importance.

- 1) Heat transfer from the moving bed to horizontal tubes.
- 2) Flow distribution and flow patterns in the moving bed.
- 3) Moving bed plugging.
- 4) Microsphere recycling rate (a function of microsphere attrition rate).
- 5) Evaluation of pump capacity and power requirements.
- 6) Pump shaft seals.
- 7) Internal surface erosion.
- 8) Lithium oxide-water corrosion damage (from tube leaks).
- 9) Fluctuating DNB or dryout in horizontal steam generating tubes.

3.0 STUDIES OF BASIC MOVING BED PARAMETERS

This part of the R&D Program would evaluate the basic moving bed parameters which are the basis for many of the engineering calculations upon which the fusion reactor NSS is based. The most important of these type of parameters are the:

- effective density,
- effective viscosity (or drag coefficient),
- microsphere attrition rate, and
- effective static bed thermal conductivity.

These properties directly or indirectly effect all of the problem areas listed in Section 2.0, except Numbers 6, 8, and 9. Tests designed to evaluate the properties listed can be of a bench scale size using relatively simple apparatus. Such apparatus for evaluating the bed material properties in the Archimedes lift could be of the circular torus design as shown in Section 3.2.3 of Reference 1 and duplicated here as Figure 1. Attrition data can be obtained from such a torus if it is made of the "lift" design material and heated to expected operating temperatures. Average material density and viscosity or drag coefficient can be obtained from a similar model fabricated from plexiglass or other transparent material. Such a visualization test would, however, require the assumption that temperature has no effect on the properties being studied. Such information can be used to determine pump power requirements and pump capacity.

The moving bed experiences two somewhat different types of flow in the nuclear steam system: a) flow by falling through the fusion reactor, steam generator and component connecting pipes, and b) flow by falling and lifting in the Archimedes pumps. There may be sufficient difference between the two flow patterns to require a second test to evaluate the effective density and viscosity (or drag coefficient) under the falling conditions. Such a test might involve passing the actual or simulated bed material through a long straight tube and then measuring the drag on the tube and the tube inventory to determine the effective viscosity (or drag coefficient) and density (see Figure 2).

FIGURE I
CIRCULAR TORUS TEST APPARATUS SKETCH.

$$P_B = \frac{W}{\pi \alpha r^2 R}$$

FROM REFERENCE *1
SECTION 3.2.3-1

FIGURE 2

FALLING BED DRAG TEST

$$\rho = 4W / \pi D^2 l$$

$$\frac{F_{\text{DRAG}}}{\text{AREA}} = 2K \Delta x / \pi D l$$

The effective static bed thermal conductivity may play an important role in heat transfer to and from the bed and within the bed itself. Standard testing apparatus and procedures have been defined to perform such tests (ASTM Standard C177).

Once the basic bed parameters are known, the validity of standard engineering equations to evaluate system drag, erosion, etc., can be checked experimentally to insure applicability in the case of a moving bed of particles simulating a fluid. Once again, bench scale tests might be applicable given that the scaling laws produce only a small error or produce a correctable error.

4.0 STUDIES OF SYSTEM-RELATED PARAMETERS

Once the basic bed properties have been evaluated, the secondary system parameters, such as flow patterns and heat transfer coefficients, can be experimentally determined. These tests are, in general, longer lead-time items requiring larger equipment than the Section 3.0 tests. Several possible tests of both small and large scale are presented below. These are not intended to be limiting cases but examples of a wide range of tests.

4.1 Small Scale Tests

Component material erosion tests can be performed on the same type of apparatus as the sphere attrition test. In general, erosion depends upon velocity and mass flow of particles per unit area of surface. Thus, the torus test should be adequate to at least generate preliminary data for the entire fusion reactor NSS.

Limited flow-related tests can be performed on bench scale apparatus. The angle of repose of the solids could be determined this way by simply pouring the Li_2O material into a container and determining the maximum angle from the horizontal. A further piece of flow information can be obtained in a 2-D, thin model consisting of a box with a hole in the center of the bottom filled with granular material. As material flows out, a boundary with a characteristic angle (usually different than the angle of repose) will develop between the moving particles and stationary particles. This is similar to the test proposed by Zenz in Section 3.2.3 - 6 in Reference 1.

Some limited small scale heat transfer work with a moving bed moving over a horizontal tube can be performed. However, past experience and large heat losses relative to test section heat inputs have indicated that such data may be of little value.

Finally, some "general" moving bed flow characterization tests can be performed. For example, a plastic 2-D model of a configuration similar to that on Figure 3A will give qualitative information about how well the moving bed simulates a liquid. Figure 3B shows a 2-D plastic model which would indicate, qualitatively, how the moving bed might react in a cross-flow heat exchanger similar to the proposed steam generator.

FIGURE 3A - 2-D FLOW CHARACTERIZATION APPARATUS

FIGURE 3B - 2D TUBE BUNDLE FLOW STUDY

4.2 Large Scale Tests

To achieve meaningful results for the moving bed concept, many of the development related R&D tests can only be performed on large test facilities which are or approach full size. This is especially true of flow and heat transfer modeling.

DNB may prove to be a serious problem in horizontal tubes on the water side of the steam generator. Even with high purity water, a DNB or dryout can fluctuate back and forth inside a horizontal tube and result in tube damage or failure by thermal stress fatigue. At the present time, DNB tests are run at full scale in order to obtain the most reliable data. Similarly, the most accurate heat transfer information comes from full scale modeling (especially in moving and fluidized beds) (see Section 4.2.1).

Flow studies will eventually be required to guarantee that proper flow distribution of the microspheres to the heat transfer surface occurs. These tests of component inlets and outlets should be operated very close to full scale in order to obtain reliable results from the flowing solids system or a two-phase gas solids system.

Because of the large size of the Archimedes pump seals, the hard vacuum required, the high temperatures, and the long life requirements, full scale testing of the seal arrangements is highly probable. Such tests would require simulation of actual conditions to insure data reliability.

The potential problems with contact between lithium oxide and water will necessitate two types of tests which should be performed early in the NSS development program. The first type of test is necessary to characterize the types of damage that would be expected from a tube leak. Lithium oxide and samples of the NSS component materials would be placed in a small test apparatus and brought up to the expected operating conditions. Water would be injected into the model to simulate a tube leak. The types of corrosion damage would then be recorded.

The second type of test would evaluate the system reaction to, and damage from a tube leak. A large pressure vessel could be modified to simulate the steam generator at the operating conditions, and a tube leak simulated. The moving bed reaction to the corrosive environment can be evaluated along with the effectiveness of the Li_2O removal technique.

4.2.1 Heat Transfer

In order to design the steam generator for the fusion reactor NSS concept, extensive data will be required in two areas:

- Local heat transfer coefficient from the moving bed to the tube outside surface.
- DNB in horizontal tubes.

Though time and funding constraints of this project precluded a thorough literature search in either of these areas, related work on the horizontal tubes in the fluidized bed program and some brief literature scanning indicated that there is virtually no reliable, readily available data for heat transfer from a cross-flow moving bed to horizontal tubes and that there is very little hard data readily available for fluctuating DNB in horizontal tubes. The result of this is that any program developing the fusion reactor NSS, as outlined in Reference 1, will have to develop all of the data required for both DNB and local heat transfer coefficients.

As indicated in Section 4.2, full scale tests will be required to perform either of these studies and obtain significant results.

Fluctuating DNB in horizontal tubes even in the presence of perfect water chemistry may eventually cause a tube to fail by thermal fatigue. To evaluate this potential thermal fatigue problem, a stress analysis is necessary. Such an analysis will require information about the frequency and amplitude of the oscillating wall temperature during the fluctuating sustained DNB. Thus, a sustained DNB test will have to be run for the final NSS generator configuration. A single tube of the same I.D. as that in the proposed steam generator could be used in a flow loop, similar to the Boiling Heat Transfer Facility at ARC, to generate DNB transient data over the entire set of operating conditions expected in the NSS generator.

To obtain the local heat transfer coefficients for the steam generator, a complete cross-section of the entire generator may have to be tested. The length of the section will not only depend on the accuracy of the data required, but also on the final design of the generator with the test section covering all of the different cross-section configurations present in the final design. A moving bed of lithium oxide spheres would have to be heated and circulated in a large loop to maintain the correct moving bed flow rate and temperature condition over the tube bank.

A less accurate method of obtaining the heat transfer coefficient data would be to neglect the tube bundle effects and to only use one tube in the test. There is a large amount of uncertainty involved in this procedure and, thus, it is not recommended.

Based on this discussion, the collection of the heat transfer and DNB data will be a costly and time-consuming activity which will have to be part of any NSS development program which employs horizontal steam generator tubes.

5.0 CLOSURE

This letter completes the last half of the ARC review of the Fusion Reactor Nuclear Steam System Concept Contract. The first half of this project involved the general review of the steam system concept and was reported in Reference 2. This reference is included as Attachment 1.

6.0 REFERENCES

- 1) D. C. Schluderberg, Li₂O Microsphere Fabrication, Monthly Letter Report #4 for the Period Ending August 31, 1977, B&W Contract No. CRD1007, September, 1977.
- 2) J. B. Kitto, "ARC Comments on the Moving Bed Fusion Reactor Steam Generator Concept", Letter to D. C. Schluderberg, File No. 5535, August 30, 1977.

A handwritten signature in dark ink, appearing to read 'J. B. Kitto', with a stylized flourish at the end.

J. B. Kitto

1md
Att.

Research and Development Division

Alliance Research Center Alliance, Ohio 44601

D. C. SCHLUDERBERG - PRODUCT DEVELOPMENT,
NPGD, LYNCHBURG

J. B. KITTO - STEAM GENERATOR TECHNOLOGY SECTION, ARC

RC-1 (rev. 1)

File No. 5535-01
or Ref.Date
AUGUST 30, 1977Subj. ARC COMMENTS ON THE MOVING BED FUSION REACTOR
STEAM GENERATOR CONCEPT

This letter to cover one customer and one subject only

1. INTRODUCTION

On August 29, 1977, a meeting was held at ARC to discuss the lithium oxide (Li_2O) moving bed steam generator concept being developed by NPGD for ERDA. In attendance were:

H. R. Carter	T. A. McNary
J. H. Kidwell	W. J. Oberjohn
J. B. Kitto	G. B. Watson

The following summary of the comments made during the discussion of the concept, as shown in Figures 1 and 2, is broken down into three areas: chemical corrosion, heat transfer and flow, and general design.

2. DISCUSSION SUMMARY2.1 CHEMICAL CORROSION

If water from the steam generator leaks into the Li_2O moving bed, the Li_2O will react with the water to generate a caustic corrosive agent ($\text{Li}_2\text{O} + \text{H}_2\text{O} \rightarrow 2\text{LiOH}$). The presence of this agent in the shell side of the steam generator will result in the chemical attack of the proposed croloy 2-1/4 tubes as well as the baffle or spacer plates. If there is a delay in removal of the large lithium oxide inventory in the generator, there is the possibility that significant corrosion damage and baffle plate plugging might occur in the generally inaccessible shell side of the generator. This eventually could result in the need for replacing the entire steam generator.

cc: H. R. Carter
J. H. Kidwell
J. B. Kitto
M. Lukasik - CRD
T. A. McNary
W. J. Oberjohn
G. B. Watson
C. P. Welch

In addition, steam may leak quickly through the high vacuum moving bed into the other major components of the system: the fusion reactor and archimedes pumps. Damage and the resulting repair work in these parts of the system would require significant downtime as well as dollars.

Due to the potential for significant damage, it was recommended that design alternatives which did not involve Li_2O to water heat transfer be looked at. An intermediate flow loop containing a fluid which will not react with Li_2O or water is one alternative while heat pipe technology might be another.

In the event that the steam generator can be quickly isolated from the rest of the moving bed flow loop, a tube leak would only cause damage in the steam generator. In this case, some damage could be avoided if a more corrosion resistant material were used in the generator. This is particularly true of the tubes.

2.2 FLOW AND HEAT TRANSFER

High heat transfer coefficients are assumed from the moving Li_2O bed of small spheres to the generator tubes ($\sim 100\mu\text{m}$). However, the moving bed will be operating in a vacuum while the data which we have seen has all been obtained in a gas at atmospheric pressure or higher. This could result in lower heat transfer coefficients in the bed than anticipated. In addition, it was questioned how the moving bed would mix in the vacuum environment. If the bed is not continually mixed, the overall heat transfer coefficient may drop significantly as the spheres near the heat transfer surface are cooled and not replaced by hotter particles from the main moving bed stream. Spiral external fins on the steam generator tubes would help promote the required heat transfer by causing continuous mixing which would bring "fresh" hot spheres to the tube or fin surface. Obviously, particle dynamics in the moving bed will be very important in determination of the heat transfer rate.

The flow distribution of the lithium oxide spheres at the steam generator inlet could present a problem. In the design shown in Figure 1, the particles will have a tendency to flow along the outside of the "C" shaped steam generator and follow the steam generator wall. This is likely because of the high resistance that the particle flow will experience when crossing the tube bundle (in cross flow) to reach the inside of the "C" shaped generator. It was felt that baffles at the inlet may not be sufficient to distribute the flow, at least with the current generator inlet geometry. A baffle ring with a preferential broach pattern placed in the vertical portion of the tube bundle might help, but there is a limit on the size of the differential such a plate will provide without causing pluggage.

Flow distribution on the water side may also generate a problem. Two-phase flow in the tubes of the bundle could result in flow instabilities within the bundle itself. In addition, a maldistribution of flow is also likely. These problems can be overcome by providing flow orifices or restrictors at the entrance to each tube. The higher resistance will prevent instabilities and promote good distribution.

Because of the uncertainties in the fluid particle flow distribution, tests will be required. Such tests may have to be full scale in size, at least at the inlet and outlet regions of the generator.

2.3 GENERAL DESIGN COMMENTS

The following general comments were generated during the discussion of the overall system.

- o B&W has no experience with archimedes pumps at this time. It might be advantageous to contact such a pump manufacturer for operating information.
- o The pump seals may present a significant problem, especially in trying to maintain a vacuum, and retaining the helium atmosphere over the extended period of time during which uninterrupted plant operation will be required.
- o The inside surfaces of all components should be designed to prevent Li_2O retention when the system inventory is removed. This is especially important in the case of a steam generator tube leak.
- o Li_2O leakage is to be expected at the top steam generator baffle as well as at the bottom. Thus, a lithium oxide removal system should also be designed at the top of the "C" shaped steam generator.
- o The baffles in the top and bottom of the steam generator should be rotated to a vertical position to permit ease of construction and to minimize system stresses.
- o The attrition of the Li_2O spheres may be a critical problem for the system. Generation of large quantities of fines in the system could clog portions of the system and prevent the correct or desired flow patterns.

3. IMPORTANT AREAS OF CONCERN

The two most important comments brought out during the meeting were the possible corrosion damage which could result from a tube leak, and the flow distribution problems which could greatly influence the steam generator performance. Both of these areas could require significant changes in the steam generator system design. Thus, further study in these two areas is required before the design concept can be brought to its final stages.

4. CLOSURE

Since the time of the review meeting discussed above, I have been informed by D. C. Schluderberg that significant changes in the system design have been made. The design changes generally outlined by D. Schluderberg in his August 29, 1977 telephone conversation with me make some of the details of the comments above no longer applicable. However, the general problem areas pointed out in Section 3 still apply and must be further evaluated.

This letter completes the first half of the work requested of ARC for the Fusion Reactor Steam Generator Concept Contract. The second half of the work requested involves the review of R&D, which should be proposed for further work in the development of the moving bed fusion reactor steam generator concept.

If comments are required on the revised design as described by D. C. Schluderberg, an extension in the scope of the work requested is required. Drawings of the revised system will be needed for such a review as well as funds for the additional man-hours which must be spent. Such an additional review of the new design will depend upon the schedule constraints of the project and the manpower constraints of the Steam Generator Technology Section.

J. B. Kitto

JBK/jls

FIGURE 2

FIGURE 2

FIGURE 1

FIGURE 1

REVISIONS

REVISION NO.	DESCRIPTION	DATE	APPROVAL	M F
1	REVISED STEAM GENERATOR PIPING ADDED SEPERATOR DRUM	10/3/77	D.C.S.

THIS DOCUMENT WAS PREPARED AS AN ACCOUNT OF WORK SPONSORED BY THE UNITED STATES GOVERNMENT. NEITHER THE UNITED STATES NOR THE U.S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION NOR ANY OF THEIR CONTRACTORS, SUBCONTRACTORS OR THEIR EMPLOYEES:

- MAKE ANY WARRANTY OR REPRESENTATION EXPRESS OR IMPLIED, WITH RESPECT TO THE ACCURACY, COMPLETENESS, OR USEFULNESS OF THE INFORMATION, APPARATUS, METHOD, OR PROCESS DISCLOSED HEREIN MAY NOT INFRINGE PRIVATELY OWNED RIGHTS; OR
- ASSUMES ANY LIABILITIES WITH RESPECT TO THE USE OF, OR FOR DAMAGES RESULTING FROM THE USE OF, ANY INFORMATION, APPARATUS, METHOD OR PROCESS DISCLOSED HEREIN.

FIG. 2-2
680-3133

ALT ARR'G'T OF NSS SYSTEM AND
ARCHIMEDES LIFT PUMPS FOR 1500
MWt Li₂O MOVING BED TOKAMAK
FUSION POWER PLANT

THIS DRAWING IS THE PROPERTY OF
THE BABCOCK & WILCOX CO.
POWER GENERATION GROUP
AND IS LOANED UPON CONDITION THAT
IT IS NOT TO BE REPRODUCED OR COPIED
IN WHOLE OR IN PART, OR USED FOR
FURNISHING INFORMATION TO OTHERS,
OR FOR ANY OTHER PURPOSE DETRIMENTAL
TO THE INTEREST OF THE BABCOCK
& WILCOX COMPANY AND WILL BE
RETURNED UPON REQUEST.

DWN. BY G.T. EDWARDS
CHKD. BY D.C. Schludermann
PASSED BY
DATE 8/24/77

SCALE 1/16" = 12"

DWG. NO. EDO 82477 B
REV. 1

THIS DOCUMENT WAS PREPARED AS AN ACCOUNT OF WORK SPONSORED BY THE UNITED STATES GOVERNMENT. NEITHER THE UNITED STATES NOR THE U.S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION NOR ANY OF THEIR CONTRACTORS, SUBCONTRACTORS OR THEIR EMPLOYEES:

- MAKE ANY WARRANTY OR REPRESENTATION EXPRESS OR IMPLIED, WITH RESPECT TO THE ACCURACY, COMPLETENESS, OR USEFULNESS OF THE INFORMATION, APPARATUS, METHOD, OR PROCESS DISCLOSED HEREIN MAY NOT INFRINGE PRIVATELY OWNED RIGHTS; OR
- ASSUMES ANY LIABILITIES WITH RESPECT TO THE USE OF, OR FOR DAMAGES RESULTING FROM THE USE OF, ANY INFORMATION, APPARATUS, METHOD OR PROCESS DISCLOSED HEREIN.

DETAIL-A
SCALE: 1/2"=1'-0"

FIG. 2.3-1

DESIGN BY: M. CALLOWAY	HORIZONTAL TUBE STEAM GEN. ARRGT. FOR Li₂O MOVING BED TOKAMAK FUSION POWER REACTOR	BANKOCK & WILCOX CO. <small>DESIGN, ENGINEERING, CONSTRUCTION, MAINTENANCE, AND REPAIR OF INDUSTRIAL PLANTS AND EQUIPMENT. 10000 WILSON AVENUE, CHICAGO, ILL. 60642</small>
CHECKED BY: D.C. SHERIDAN		
APPROVED BY:		
DATE: 680-3133		
DO NOT SCALE - USE DIMENSIONS ONLY		SCALE: 3/4"=1'-0" REV: ED092877F 0