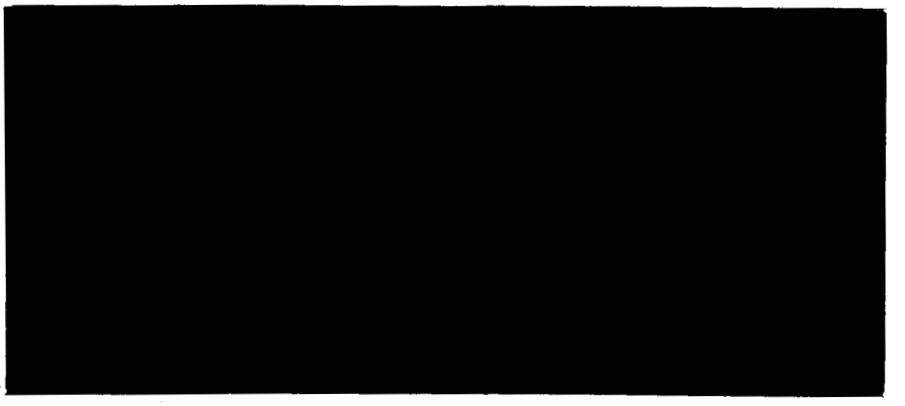

THERMO ELECTRON

ENGINEERING CORPORATION

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Report No. TE84-65

THIRD MONTHLY PROGRESS REPORT FOR THERMAL ENERGY STORAGE/CONVERTER PROTOTYPE DESIGN, FABRICATION AND TESTING

Contract No. 950976/NAS7-100

3 May through 2 June 1965

by

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1. INTRODUCTION

This report represents the Third Monthly Progress Report of the work being performed under Contract No. 950976/NAS7-100 with the Jet Propulsion Laboratory.

The objectives of this program call for the design and fabrication of one thermal energy storage feasibility model. This model shall include an electron beam weldment sealed rhenium capsule containing 3BeO-2MgO oxide, one thermionic converter of the Series VIII or TE-100 design of JPL Contract No. 950671 modified to accommodate the capsule, capsule heater, and thermal shielding.

The model shall be tested in the presence of a JPL representative for not less than 16 cycles at 50°C above and below the melting point of the oxide.

2. SUMMARY

During the month of May purification of MgO and BeO powders was continued, and the vacuum firing procedures used resulted in an overall decrease in the impurity levels of the two "as received" MgO powders from Fisher Scientific and Morton Chemical. Two additional 3BeO-2MgO melts of unpurified oxide were made using the MgO from the two different suppliers. These two melts as well as the first melt (made with Morton MgO) were made in vacuum and subsequent determination of the BeO/MgO ratio indicated that the required stoichiometry was retained. The melt using Fisher MgO has a very clean external appearance in comparison to the other melts, and is presently being analyzed.

An electron bombardment heater was built and tested using various diameters of tungsten and rhenium wire. Fifteen mil tungsten appears to be the best choice for the feasibility model heater. Under similar operating conditions rhenium wire did not demonstrate the same life capability as tungsten.

A full scale version of the thermal shield assembly was built for evaluation. Since thin rhenium sheet which is on order for shielding was not available, tantalum was used in its place on the inner surfaces. Preliminary fabrication and ordering of all parts required for the test set-up was accomplished during this reporting period.

With the exception of the rhenium sleeve for the converter which is on order and due for delivery early in June all parts have been built, and are available for converter fabrication. All the material required for the container is either on hand or due early in June. Since rhenium sleeves have not been previously used on Set converters, a component test was made using an available (larger diameter) rhenium sleeve and a niobium flange. The joint was made in the electron beam welder. Subsequent cycling of the assembly to operating temperature demonstrated that this design is quite sound for the model.

3. PURIFICATION OF MgO AND BeO POWDERS

Magnesia and beryllia with which to make the oxide slug have been fired at high temperature by heating uncompacted oxide in a metal bucket in vacuum. An unreactive container is required, and rhenium is found to fulfill this requirement adequately. Purification is effected by evaporation of volatile impurities, which condense in a cooler part of the system. It is therefore necessary for the whole mass of oxide and its container to be isothermal, and hotter than some nearby part of the system, which can act as a condenser.

Electron-bombardment heating of the bottom of the right cylindrical container was found to leave the upper open end much cooler, and to be rather inconvenient to manipulate. Radio frequency induction heating was therefore utilized. However, if the container was supported on a pedestal in the usual manner, the center of the bottom was cool owing to heat loss by conduction down the pedestal, and the center of the top was cool owing to radiation loss from the open top of the container. A much closer approach to isothermal conditions was obtained by supporting the container on tantalum wires just above the pedestal, and closing the top with a loose fitting lid of rhenium. See Figure 1. This eliminated the dark patch on top of the oxide, which had presumably been caused by condensation of contaminants there. Upon further heating, a dark deposit was found on the underside of the lid.

All of this work was complicated and slowed by the tendency of the dry uncompacted oxide to "explode" and spatter all over the vycor and baseplate during the early stages of evacuation and heating. Unfortunately, no way of accelerating the process is obvious. To use a larger diameter container would interfere with achieving isothermal conditions throughout the container, since the heat is developed only in the walls of the container.

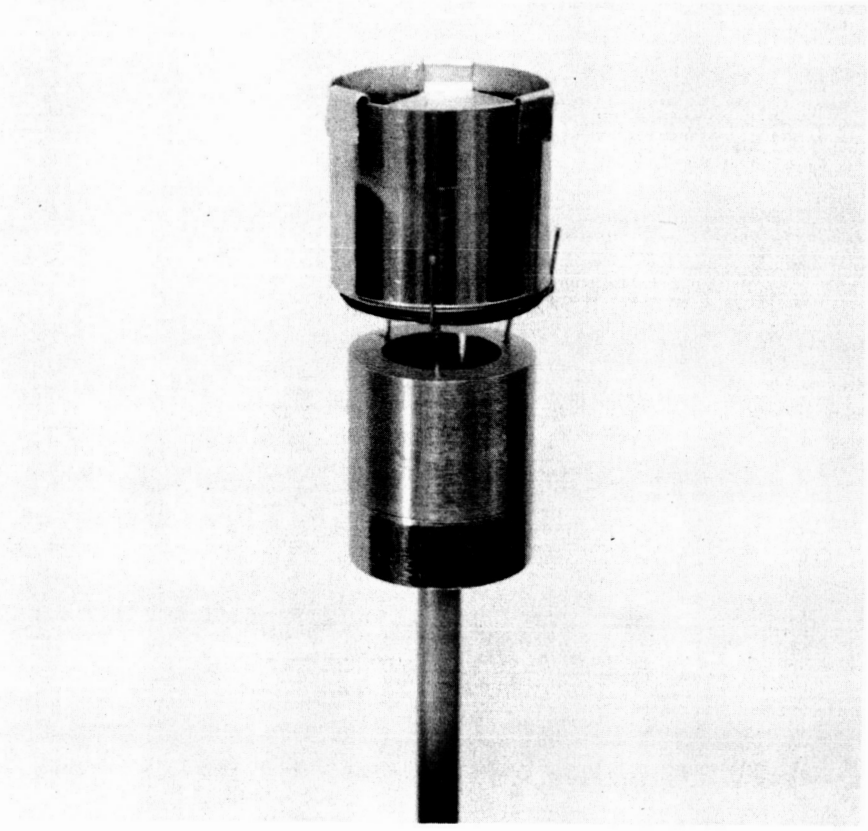

Figure 1. Rhenium Crucible Mounted for RF Heating

Most of this purification work was done with BeO, while awaiting results of analyses of two samples of MgO (from different sources) purified by firing. These results are shown in Table 1. It is seen that considerable purification of the Morton MgO with respect to Si and Ti was achieved, while the Fisher MgO lost Na, but gained Fe and some Ca, and remained cleaner overall. The earlier hypothesis that Fisher MgO lost Na on firing was therefore substantiated.

In summary, some purification of MgO was achieved. Fisher material lost a substantial amount of Na, but appeared to gain some Fe and Ca, perhaps during handling. Morton material appeared to gain small amounts of Fe and Ca too, but lost Si and Ti, while remaining less pure overall.

On the strength of this data, it was decided to prepare a melt using Fisher MgO. (Melt #3).

TABLE I

IMPURITIES IN MgO BEFORE
AND AFTER FIRING TO 2000°C IN VACUUM

	Morton		Fisher	
	Before	After	Before	After
Ca	1100	1200	280	350*
Si	650	150*	180	150
Fe	220	250	15	30*
Ti	90	40*	< 2	< 5
Re	--	30	< 10	< 10
Cu	3	5	3	4
Na	< 5	3.5	110	2*
Sr	< 0.5	< 0.5	1.5	1.0

Significant changes are indicated by *

These are quantitative spectrographic analyses, all made by TEECO's analyst.

4. 3BeO-2MgO OXIDE PREPARATION

The first melt (Melt #1, Morton MgO) made was examined[1] for impurities and to determine the BeO/MgO ratio. Spectroscopic analysis revealed that the melt had picked up Re to the extent of 0.5 - 5.0%, and had lost some iron. The only explanation that can be offered for the Re pickup is in terms of the high temperature to which this melt had been exposed (2600°C). All other impurities detected were present in a similar amount in the starting oxides.

Most interesting was the estimate of the wt BeO/MgO ratio as 1 : 1. The target ratio, corresponding to 3BeO : 2MgO, is 0.931 : 1. So despite the very high temperature obtained by this melt, little MgO had been lost by evaporation. This is felt to be very encouraging, as trouble was anticipated owing to the high vapor pressure of MgO at the eutectic temperature. However, in case it proves necessary to hold the composition of the melt very close to that of the powder mixture, plans have been made to operate RF heating in an inert atmosphere such as argon, and an appropriate piece of equipment is under construction.

A cross-section of this melt, examined under the microscope was found to contain some very large bubbles, but very few fine pores. No discernable microstructure was seen. The melt was a light gray color. This melt was made with as-received unpurified oxides early in the program. The most significant feature of this first melt was the contraction which occurred on melting. Though the container had been filled with mixed oxides, after melting there was not enough to wet the whole of the bottom of the container. This is due primarily to the contraction of low density powder to fully dense liquid.

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[1] By National Spectrographic Lab., Cleveland, Ohio

A second melt (Melt #2, Morton MgO) was made, aimed primarily at achieving a larger quantity of molten material. Sufficient unpurified oxides were blended with alcohol to more than fill the bucket, and it was filled about 70% with oxide slurry. This was warmed to drive off most of the alcohol, and then evacuated and heated to the melting point. After a minute or so at the melting point, the heat was removed and, after cooling, more of the same batch of oxide was added, dried out, pumped down, and remelted. This procedure was repeated twice more.

The resulting slug occupied about 1/3 of the container, slipped readily out of the container when cold, and solidified with a shallow rounded crater at the top, implying that it contracted on freezing. No quantitative measurement could be made because of the irregular pores in the material. It had the gray color characteristic of the first melt (both were made with unpurified oxides). It was examined for BeO/MgO ratio and spectroscopically for trace impurities.[2] The principle trace was iron, followed by silicon, and these and the other traces were present in amounts comparable with the starting oxides, but purification with respect to Ca was considerable, and some Ti was lost. No rhenium was detected, despite the discoloration of the material, and a detectability limit of 1 ppm for it. This melt was never at the temperatures reached by the first melt. The BeO/MgO weight ratio was found to be 0.947, which agrees with the target ratio 0.931 within the limits of experimental error. Again, MgO evaporation was not a significant problem.

Since air firing is a standard method of cleaning ceramics, it was applied to part of the second slug. Firing was carried out for 1 hour at 1200°C in air, supporting the sample on pure Al_2O_3 ceramics. Some cleaning of the specimen was

[2] By Skinner and Sherman, Inc., Newton, Mass.

found, and it was therefore fired for another five hours in the same conditions. This left the specimen clean, that is, of yellowish-white appearance, throughout, except for some metallic gray flecks. A further examination is now under way to determine which impurities are lost on air firing. These are presumably the ones which cause the gray color.

A third melt was made using Fisher MgO in place of Morton. This was quite satisfactory and appeared very clean and free from gray contamination on removal from the container, Figure 2. Apparently the purer Fisher oxide leads to a cleaner product when used without special purification. During melting, considerable deposition occurred on the bell-jar, implying that purification and melting both occurred. Further examination of this melt is underway.

A piece of scrap 0.02" Re sheet was included in the second melt made. It was extracted from the resulting slug, and sections were examined metallographically. Some traces of fast-etching areas and possible pores or inclusions were found along the center line of the sheet. These are not normally present in sound rhenium sheet. They are, however, found near the edge of sheared rhenium sheet. Further work is planned for placing rhenium in melts after careful polishing of the rhenium along the edges to remove traces of shearing.

The external and internal appearance of the rhenium containers used is still immaculate and they are perfectly leaktight and the polished sections show no signs of attack at the surfaces of the sheet, though this would be the first place to be attacked if there was any interaction.

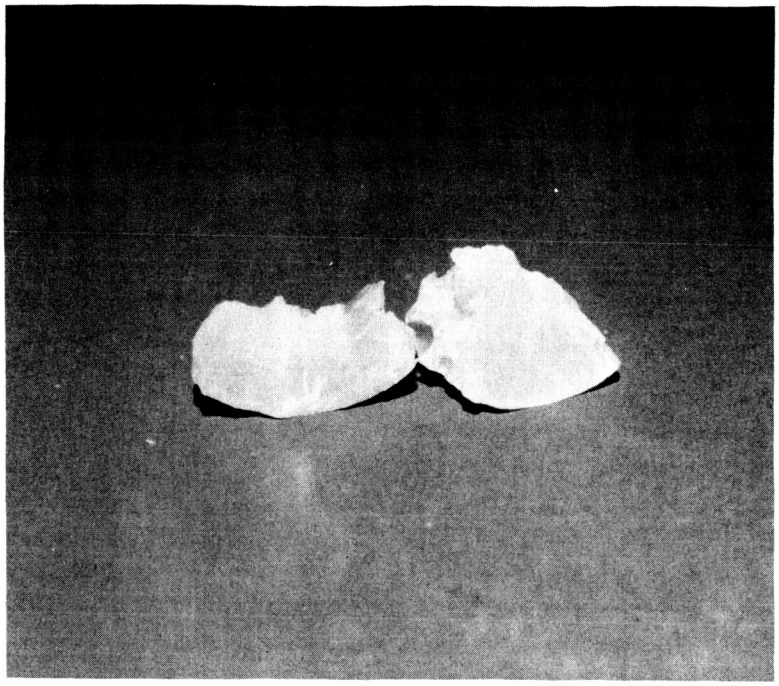

Figure 2. 3BeO-2MgO Eutectic from Melt #3

5. FEASIBILITY MODEL

To gain confidence in the proposed designs of the model's thermal shielding and electron bombardment heater, both units were built for evaluation with a dummy container.

The thermal shielding design used as many concepts as possible from both the solar and isotope generators which have performed very well. An exploded view of the major sub-assemblies used for the shields is shown in Figure 3 while the completely assembled unit is shown in Figure 4. The opening in the shields shown in the latter figure is the portion that will be positioned around the emitter sleeve. Since thin rhenium sheet which is on order was not available, tantalum was used in its place on the inner surfaces. A dummy capsule will be supported inside the assembly, and heated by the gun.

A measure of the shielding effectiveness, including edge effects, will be obtained by measuring the capsule temperature, surface temperatures, and power input. With known values of emissivity for the outer surfaces, the rate of heat loss vs capsule temperature will be calculated. A check of the heat loss from the outer surfaces will be obtained with a Leeds and Northrup thermopile.

An electron bombardment gun, Figure 5, was built and tested with six wires of .010", .015", and .020" Tungsten and .015" Rhenium in four separate tests. The .010" tungsten wire was not rigid enough, while the .020" wires required high values of starting filament current - in excess of 100 amperes. The .015" tungsten wire provided adequate rigidity without excessive filament currents. The assembly with .015" wires can be mounted, dismounted, and handled after operation. The expected life of this gun is of the order of 500 hours. A heater with .015" tungsten wires is planned for the model. Under similar operating conditions

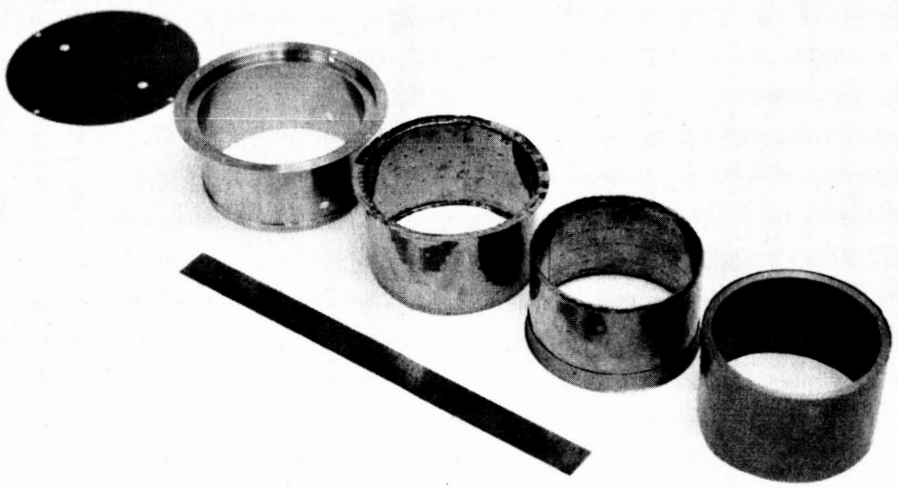

Figure 3. Exploded View of Major Thermal Shield Subassemblies

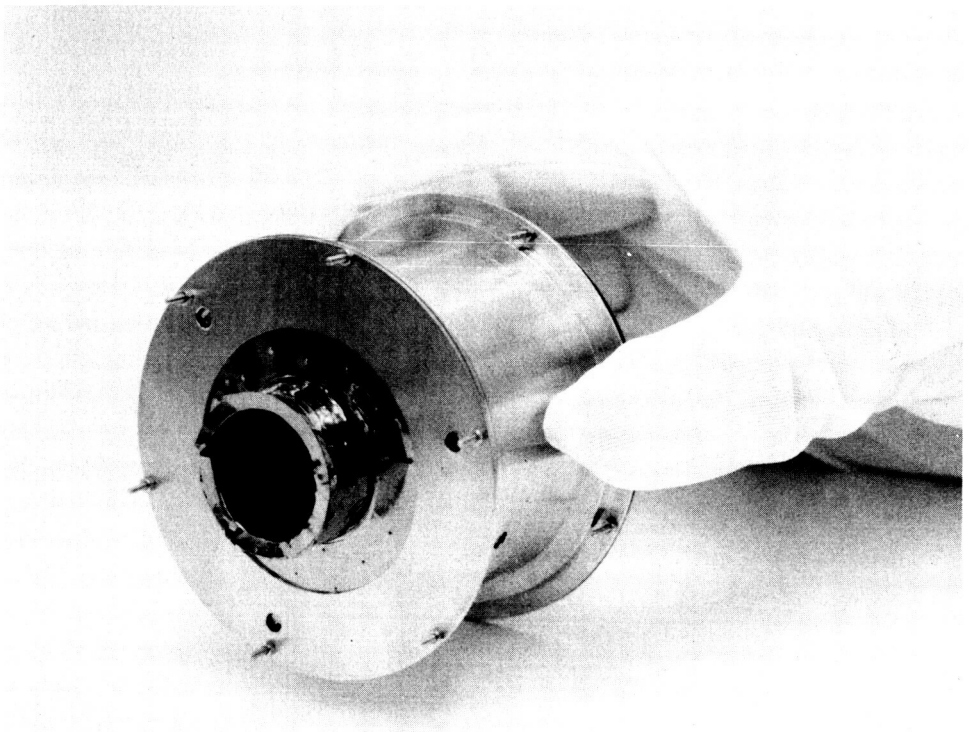

Figure 4. Thermal Shield Assembly

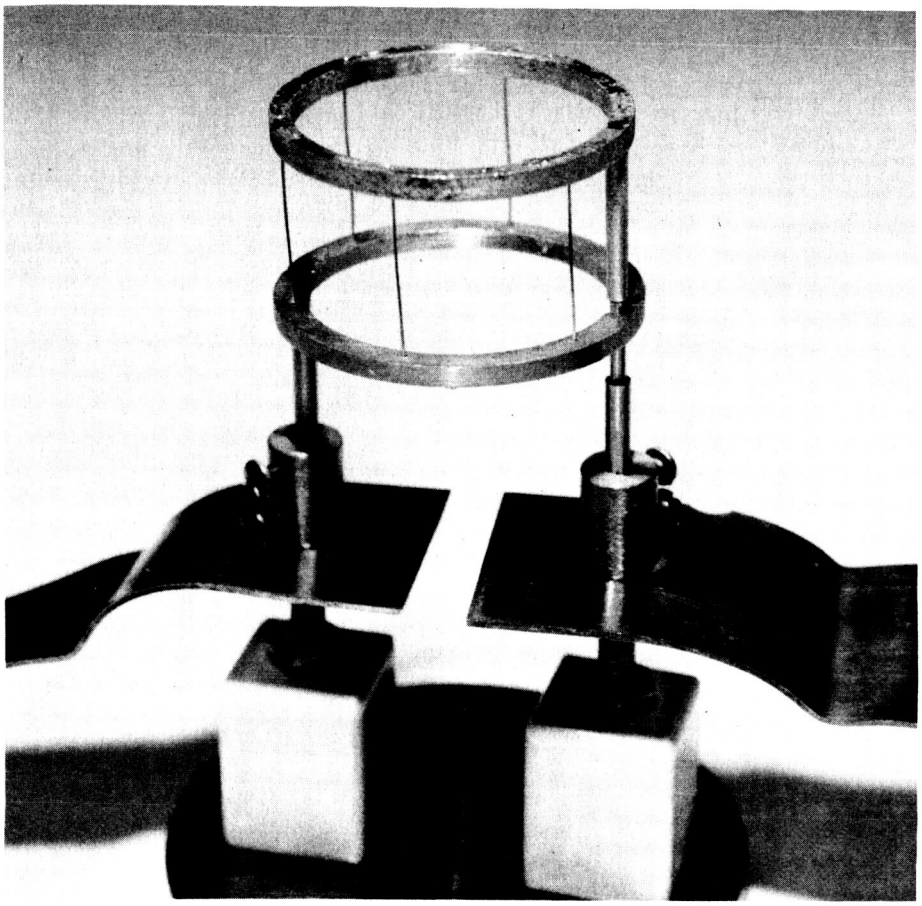

Figure 5. Electron Bombardment Heater for TES Container

rhenium wire did not demonstrate the same life capability as tungsten.

Preliminary fabrication and ordering of all parts required for the test set-up was accomplished during this reporting period.

6. CONVERTER

Since rhenium sleeves have not been previously used on Set converters, a component test was made using an available (larger diameter) rhenium sleeve similar to part 35, Figure 6, to a niobium flange, part 34. The joint was made in the electron beam welder, and is shown in Figure 7.

The joint was cycled rapidly to 900°C twenty times, and leak checked after the first and last ten cycles. The joint was leak tight both times. Subsequently, the joint was cycled to 1500°C rapidly approximately thirty times. The increased temperature was selected as a possible operating condition for another application. The joint was still sound and leak tight.

From these tests the joint design appears to be very good.

Figure 7 Niobium Flange Beam Welded to a Rhenium Sleeve