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NAVAL UNDERSEA WARFARE CENTER

An activity of the Naval Material Command

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This report presents results of an investigation to determine an underwater power source suitable for long missions at great depths. Thermal energy stored in a molten lithium salt mixture was used to generate steam that could be used to power a closed-cycle system. The thermodynamic properties of the salt, as well as the corrosive action of the salt on container materials, was investigated.

The information included here was presented to the Third Propulsion Joint Specialist Conference of the American Institute of Aeronautics and Astronautics, Washington, D. C., in July 1967. The work was performed under Bureau of Naval Weapons Task Assignment ASW 202-000/216-1/F108-01-33 during the period 1964 through 1966.

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PROBLEM

Develop an energy source that can be used to great ocean depths with minimum deterioration of performance or change in system buoyancy.

RESULTS

An analysis of various thermal storage materials indicated that a eutectic mixture of lithium hydroxide and lithium fluoride would provide relatively high specific energy for a closed thermal propulsion cycle, and would cause no buoyancy change when contained in a pressure vessel. Tests were conducted to determine the physical properties as well as the corrosive nature of the salt mixture. A study determined that Inconel 600 is the best single material for containing the molten salt under high pressures.

RECOMMENDATIONS

The salt combinations studied have many desirable characteristics, especially at temperatures below 1000° F. Above that temperature, severe corrosion problems exist. In addition, a reduction in the strength of Inconel 600 occurs when small amounts of sulphur are present. These two problems should be studied carefully before further development of a system utilizing LiF-LiOH as a thermal storage medium.

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INTRODUCTION

An ASW system now under investigation at the Naval Undersea Warfare Center (NUWC) requires an unmanned, deep-operating sonar surveillance vehicle with a very long endurance capability. Obviously, atomic energy is the only present source that could provide the necessary power throughout the complete mission. However, no applicable nuclear power source is likely to be available in the foreseeable future. As a result, an investigation of relatively short-endurance propulsion systems and energy sources was undertaken.

The investigation centered around systems capable of quick recharge or refueling from a surface vessel at sea. In this context, quick recharge was to imply a period of 6 hours or less.

In addition to quick recharging or refueling, the system should meet four other criteria:

1. It should be capable of achieving extreme depths without reduction of performance.
2. Any change in buoyancy or center of gravity should be minimal to eliminate trim problems.
3. Hazards to the crew of the tending ship must be minimal.
4. The propulsion system should be quiet, so as not to interfere with sonar systems.

With these limitations in mind, many possible energy sources were considered, and a eutectic lithium fluoride-lithium hydroxide molten salt thermal energy storage system was further studied as a prospective energy source. Some of the available energy sources are reviewed in the following paragraphs to illustrate how the LiF-LiOH eutectic compares with other energy sources.

Thermal Combustion Engines. The combustion engine has several disadvantages in deep operation. It is depth-sensitive if the products of combustion are exhausted directly overboard against high

backpressures. In fact, additional equipment is required if the products of combustion are to be pumped overboard or stored. Furthermore, trim becomes a problem if the center of gravity or buoyancy changes as the fuel is consumed, so that a ballast or buoyancy compensation system is required. Trim is particularly critical in the case of slow-moving submerged vehicles.

Batteries. Conventional storage or rechargeable batteries are more favorable with respect to buoyancy control than the combustion engine. However, the available energy per charge in batteries is quite low, the number of times that a battery can be recharged is limited, and the battery's high negative buoyancy can impose a considerable penalty if additional material is required to provide neutral buoyancy for the system.

Thermal Energy Storage Systems. Several thermal storage systems have considerably higher specific energy storage capabilities than lead-acid or silver-zinc batteries, and they also provide constant buoyancy. Although thermal storage systems do not match the theoretical performance of some thermal combustion or fuel-cell systems, the thermal storage system investigated by NUWC has the advantage of being rechargeable by means of superheated steam supplied from a surface vessel.

The thermal energy that can be stored in the eutectic compares quite favorably with other energy sources. Figure 1 is a nomograph of endurance versus specific energy for various energy sources that have been considered for underwater powerplants in small submerged vehicles, which would range from 200 to 600 cubic feet in size and require from 50 to 100 shaft horsepower. The vertical width of the band for each type of energy source is indicative of the penalty paid for the addition of buoyancy material or containers necessary to provide neutral buoyancy. The bottom line of each band represents the possible energy and endurance for each system, while the top line indicates the net available energy when buoyancy material and containers are included. On this basis, the eutectic thermal storage system applied to a Rankine cycle steam engine appears to be very favorable in comparison with rechargeable batteries. The narrowness of the cross-hatched area for the salts is indicative of the relatively small penalty incurred with their use when neutral buoyancy is required.

Fuel cells would appear to provide the greatest endurance, based on specific fuel consumption only. However, space requirements for auxiliary equipment would appear to be too great for smaller vehicles,

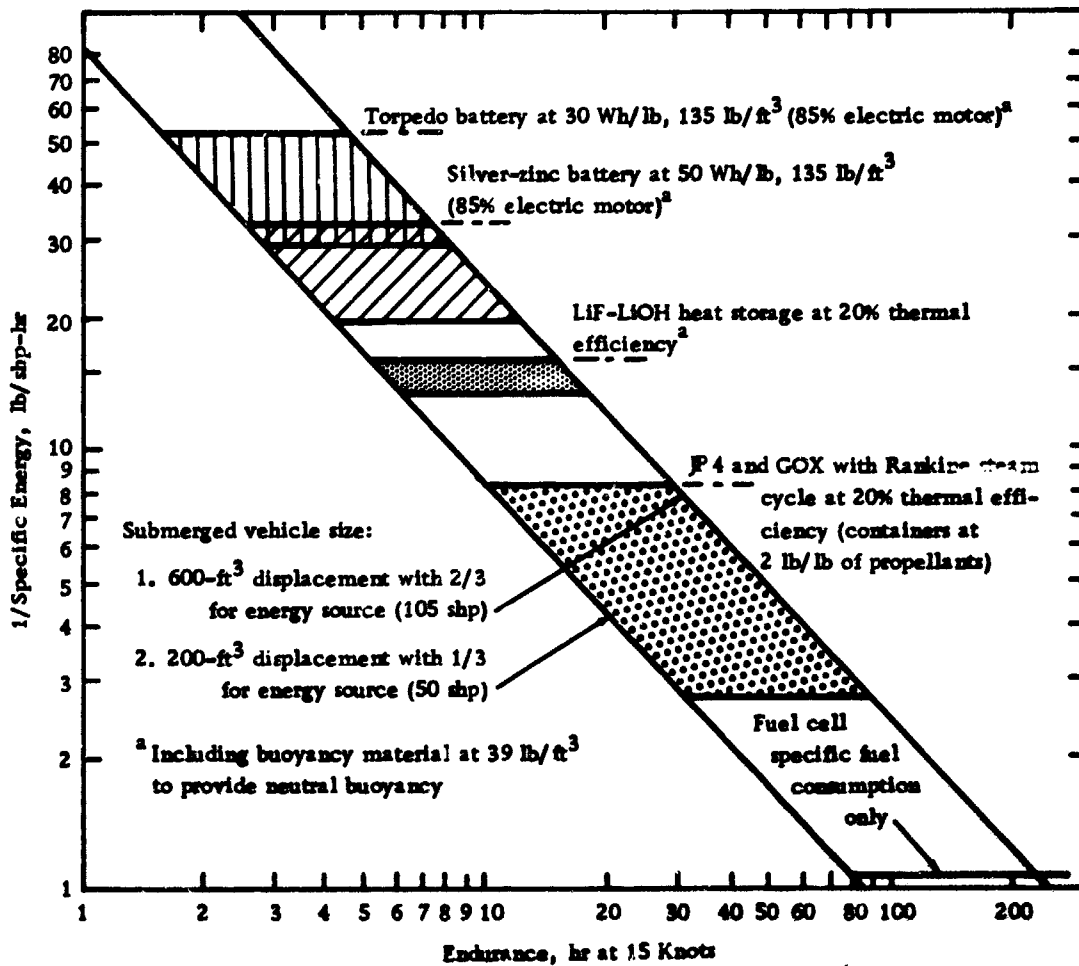


FIG. 1. Submerged Vehicle Endurance With Various Energy Sources.

and there are many problems in an undersea environment that must be overcome before the fuel cell can be seriously considered for small submerged vehicles.

Proposed System. The proposed propulsion system, which uses thermal storage as the energy source, is basically a Rankine or closed-cycle steam system with boiler tubing embedded in eutectic salts. A much simplified schematic is shown in Fig. 2. The boiler tubing is used both to extract the heat from the salts by generating steam and to recharge the eutectic salts with superheated steam from the tending vessel. Electrical heaters were not chosen for use in the charging cycle because they would add considerable extra weight. The boiler tubing was investigated for use as possible resistance elements for electric heating, but excessive corrosion

occurred because of the conductivity of the salt and the resulting electrolytic action.

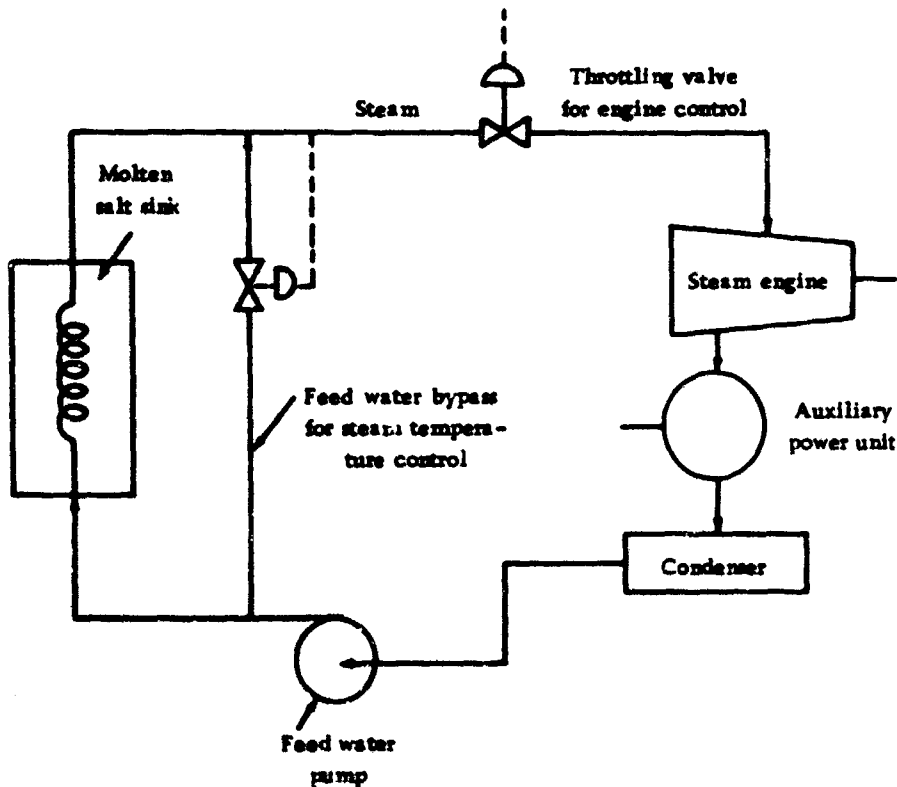


FIG. 2. Simplified Flow Diagram for a Molten Salt Thermal Energy Propulsion System.

Another advantage of a molten salt system is that the Rankine cycle used would be applicable to a nuclear thermal system in the event that nuclear power sources become available for future small submersible applications.

Several propulsion systems using a molten salt for thermal energy storage have been investigated by other laboratories for underwater vehicles and various missions. In general, these systems would provide much greater specific energy per charge than conventional storage batteries, and would also provide constant buoyancy for deep operation. However, most of these systems used either lithium hydride or lithium fluoride as the energy storage medium, and each has several undesirable features. Lithium hydride, which has the highest energy density of the lithium salts, was not considered satisfactory for three reasons:

1. It is violently reactive in contact with water, and hence is a hazard to personnel during handling.
2. It has a 16-to-19% shrinkage factor during fusion, which might cause severe stresses within the heat exchanger when cycled repeatedly.
3. It has a melting point of 1256° F, which makes recharging with ship's steam difficult.

Lithium fluoride was discarded as a sole source of energy because of its high melting point (1556° F) and high negative volumetric change during freezing (25%).

Lithium Hydroxide, another lithium salt investigated, has a more favorable melting point of 884° F and also a low volumetric change during freezing. The volume change is unusual, in that lithium hydroxide expands 3.4% on freezing, which is similar to ice. Although the volumetric change of lithium hydroxide is low in comparison with other salts, there was apprehension that difficulty might be experienced in the design of a vessel and heat exchanger to withstand possible stresses during repeated cycling. To counteract this expansion, various amounts of lithium fluoride were added to the lithium hydroxide. When 20 to 30% lithium fluoride by weight was added, practically no change in volume was noted, and the melting point of the mixture was 800° F. This led to the discovery that the point of no volume change corresponds nearly to the eutectic point of the mixture, and would provide a salt with both a low volumetric change and a low melting point. The phase diagram of these materials in Fig. 3 confirmed this eutectic point, but very little information was available on the thermodynamic properties or chemical reaction of the eutectic with other materials. Therefore, before the undertaking of an extensive test program, it was decided (1) to make some preliminary tests that might reveal problem areas, and (2) to determine the feasibility of using this salt combination as a thermal storage medium.

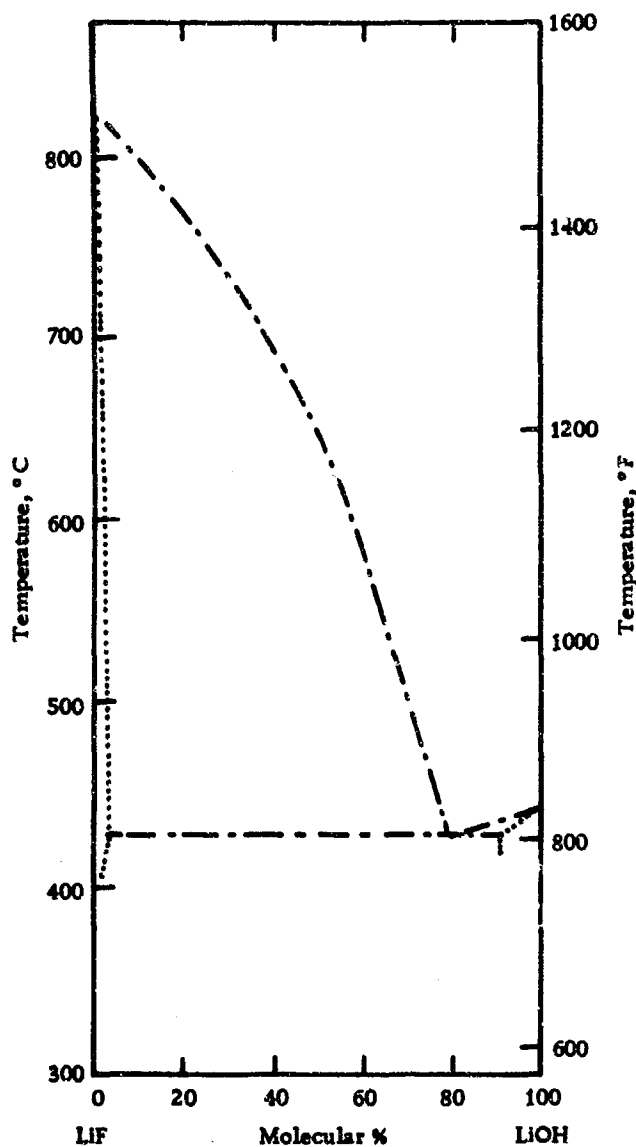


FIG. 3. Lithium Fluoride-Lithium Hydroxide (LiF-LiOH) Phase Diagram.

EXPERIMENTAL STEAM BOILER

A small heat-storage chamber was constructed to determine the practicality of generating steam from the hot salt. Also to be determined were the approximate thermodynamic properties involved and the corrosivity or breakdown of the eutectic mixture during repeated cycling in contact with available structural materials, such as stainless steel or Inconel. Figure 4 shows a schematic layout of the test

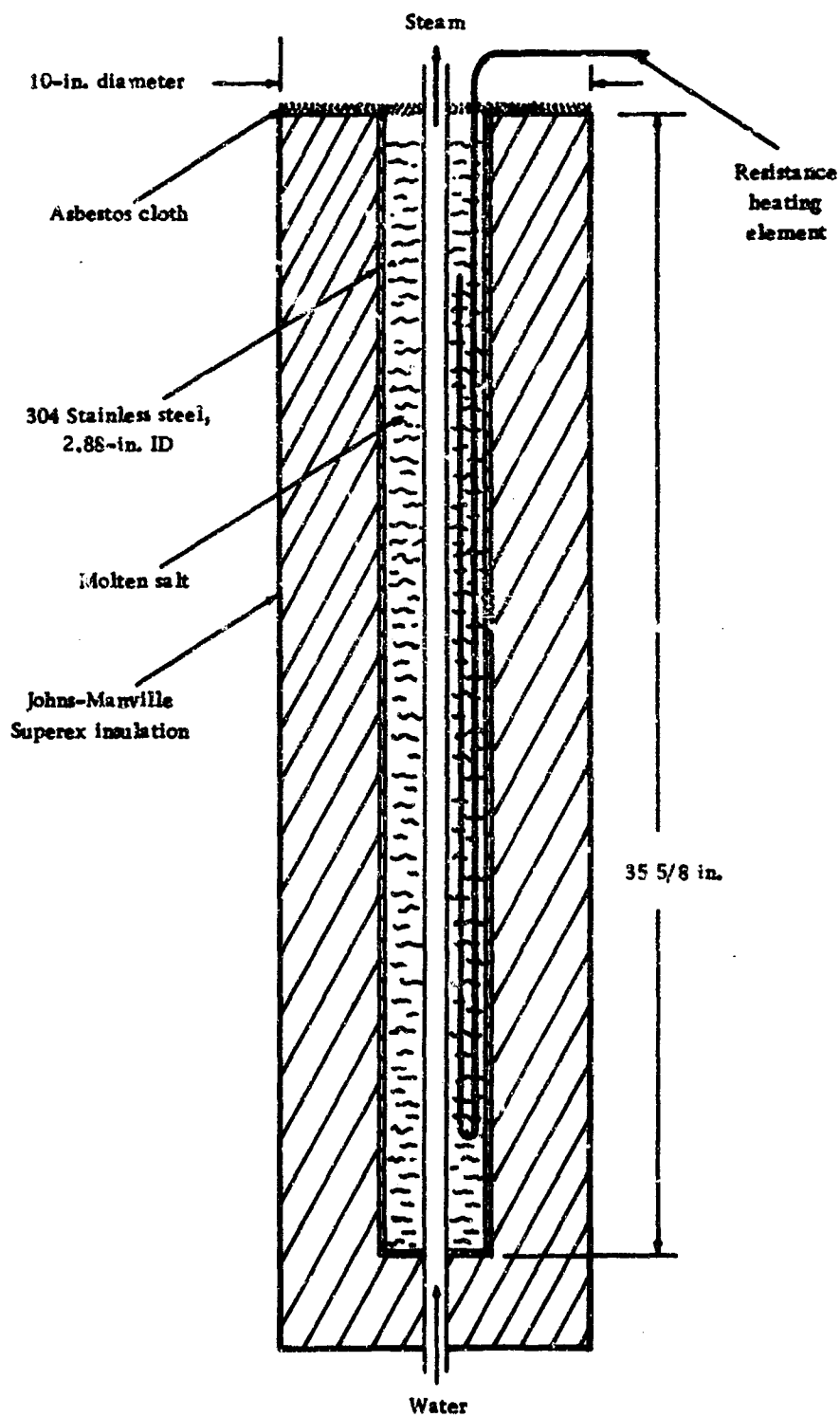


FIG. 4. Experimental Molten Salt Steam Generator; 12-lb Salt Capacity.

setup. The thermal storage container had a 3-inch internal diameter and a length of 36 inches. A heat-exchanger tube was positioned coaxially in the chamber. The tube was 0.5 inch in diameter, and had a wall thickness of 0.035 inch. Both the tube and the container were constructed of Type 304 stainless steel. The apparatus contained 12 pounds of the eutectic mixture. Inconel-sheathed thermocouples were located at various elevations in the container and the steam tube. The outer shell was encased in high-temperature insulation (Johns-Manville Superex). An Inconel-sheathed resistance heating element was immersed in the salt to melt the mixture.

After heating the salts to a molten state, distilled water was introduced into the bottom of the steam tube by a Sprague pump at pressures up to 1,000 psig. As the water flowed up the tube, it was transformed to steam by the heat absorbed from the hot salt. At the beginning of the run, the majority of the heat was extracted only from the lower part of the apparatus. As the run progressed, the heat was extracted at higher and higher locations until, at the end of the run, the top part of the salt had cooled below 700° F. A typical run record is shown in Fig. 5. Note that it is important to be able to extract heat from the salt in a stratified manner. More heat can thus be obtained from the salt, and steam can be generated at higher temperatures than would be possible if the salt cooled uniformly.

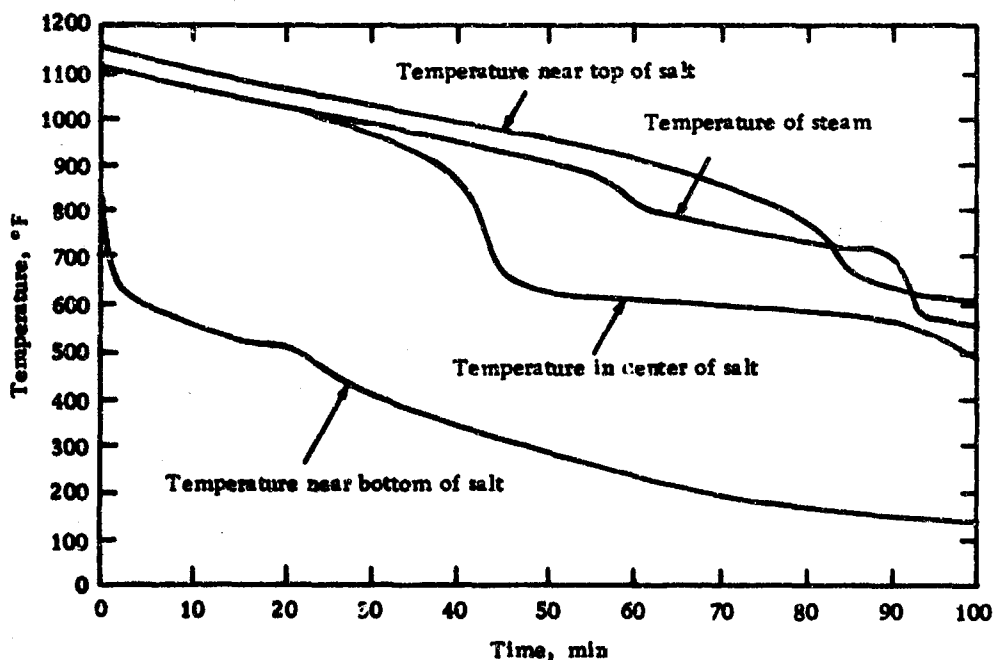


FIG. 5. Temperature-Versus-Time Cooling Curves for Experimental 12-lb Steam Generator With a Water Flow Rate of 4 lb/hr.

In one run, superheated steam was generated at temperatures between 700 and 1200° F at 1,000 psig for 1.5 hours at a rate of 4 pounds per hour; in another run, the period was 4 hours at a rate of 1.5 pounds per hour. A total flow of 8 pounds of steam was possible at low flow rates, which indicated that the available heat is approximately 1,000 Btu per pound of the eutectic salt mixture. A third test run was made, in which the steam temperature fell from 1200° F to 700° F in 17 minutes, with a flow rate of 15 pounds per hour. An additional 4 pounds of superheated steam was generated by lowering the flow rate.

CORROSION

About 30 test runs were made with the 12-pound unit, during which the salt mixture was in the molten state at temperatures from 800 to 1300° F for approximately 200 hours, and in the partially molten state during reheat for several times that long. No deterioration of the salts or failure of the steam tube, Inconel-sheathed thermocouples, or the heater exposed to the salts was detected from test data taken during this period. The tests were then discontinued, as it was believed that sufficient information had been obtained for preliminary design purposes.

The 12-pound salt container was later cut into sections. Only the bottom portion of the salt column indicated deterioration, as shown by corrosion products that had settled there. The upper portions of the column appeared to be uncontaminated. The wall of the stainless-steel tube underwent some corrosion at the upper section, where high temperatures were sustained for greater periods of time. However, most of the corrosion was on the inside surface, which was exposed to steam only. These results were considered quite favorable, especially since the material used for construction was common 304 stainless steel.

Nevertheless, subsequent tests on samples of various metals indicated that rapid corrosion may occur. It was therefore considered necessary to conduct a more extensive investigation to determine the best materials to withstand corrosion, and to determine the conditions that cause accelerated corrosion. A contract for this investigation was let to Atomic International, Division of North American Aviation, Canoga Park, California.¹

¹ DeVries, G., and L. F. Grantham. "Corrosion of Metals in the Molten LiOH-LiF-H₂O System," *Electrochemical Technology*, July-August 1967, Vol. 5, No. 7-8, p. 335.

Screening tests were made on nearly 50 different metals and alloys to determine those which were worthy of further study. Nickel, some nickel-based alloys, and the precious metals, silver, gold, and platinum proved to have the best short-term corrosion resistance. Longer-term tests of 550 hours were conducted on nickel and nickel-base alloys. The two most promising containment materials indicated were nickel and Inconel 600. The average corrosion rates during the 550-hour test were 0.027 mil/day for nickel and 0.27 mil/day for Inconel 600 at a test temperature of 1200° F.

The effect of temperature on the corrosion rate is shown in Fig. 6. As expected, corrosion increases significantly as the temperature is increased. However, it is important to note that below 900° F the corrosion rate is insignificant.

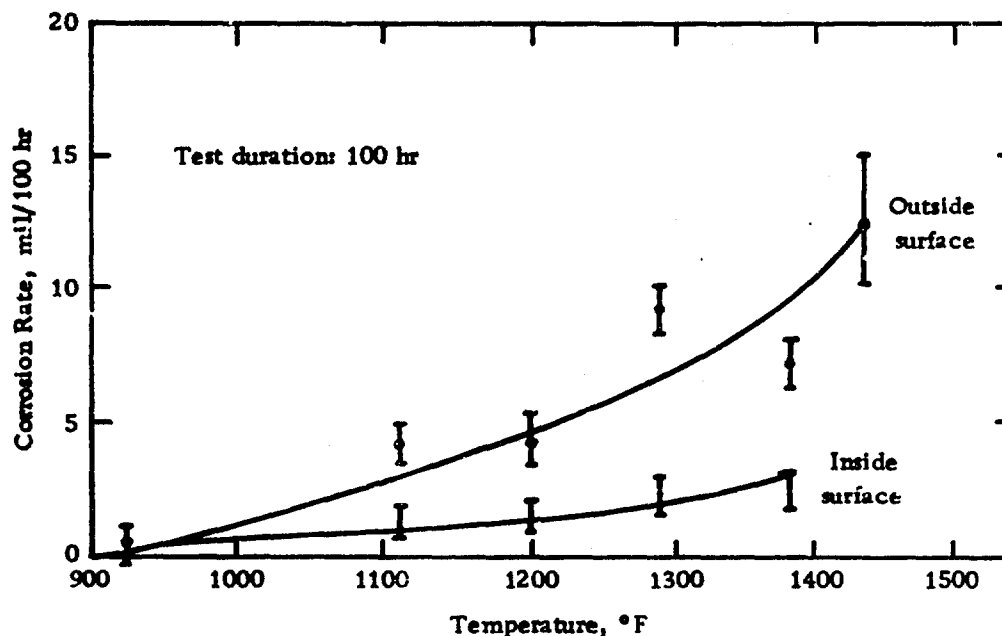


FIG. 6. Corrosion Rates Versus Temperature of a Section of 3/16-in. -OD Inconel 600 Tube in a Molten LiF-LiOH Environment.

The impurities sulphate and silica increase the corrosion rate of most container materials. It was found that small amounts of sulphate ions in the melt increased the corrosion rate significantly in all cases. For some high-iron-content alloys, dissolved silica in the melt also increased the corrosion rate. In the case of Inconel 600, however, silica was found to have little effect on the corrosion rate. This is important because many of the insulating materials contain silica, and Inconel 600 appears to be the best containment material. Sulphur-

bearing compounds in insulating materials are suspected of causing catastrophic failures of test containers on several occasions. The explanation is that nickel is subject to attack by sulphur gases at temperatures above 600 to 700° F. Reducing conditions are worse than oxidizing ones and, at temperatures above 1190° F, a eutectic of nickel and nickel sulphide forms. Being molten, the eutectic penetrates the grain boundaries and leads to disintegration of the metals.²

SALT CHARACTERISTICS

A contract was let to the Dynamic Science Corp., Monrovia, California, to determine the thermodynamic characteristics of the salt. A parallel effort was conducted in-house to determine the physical characteristics of the salt and to check the results of the thermodynamic study. Table 1 lists the basic properties of the salt, as determined by these two studies.

TABLE 1. Properties of Salt Used in Heat Storage

Composition	20% LiF - 80% LiOH (eutectic mixture)
Melting point	800°F
Volume change (70 to 1200°F)	≈ 7%
Density	90 lb/ft ³ at 1200°F 97 lb/ft ³ at 70°F
Average specific heat:	
Solid	0.51 Btu/lb/°F
Liquid	0.39 Btu/lb/°F
Heat capacity from 300 to 1200°F:	
Solid	260 Btu/lb
Fusion	500
Liquid	<u>170</u>
Total	930 Btu/lb

An important feature of the properties is that the salt density is only 40% greater than that of water. Hence the space-weight penalty paid to make the system neutrally buoyant is not as great as for other energy sources, for which containers, pumps, plumbing, pressure vessels, and other heavy components require large quantities of flotation material to make the system neutrally buoyant.

² International Nickel Co., Inc. Nickel and Nickel Base Alloys. Huntington, Virginia, International. (Technical Bulletin T-13.)

Enthalpy. Figure 7 gives a better idea of the enthalpy distribution for the salt. It is significant to note that 87% of the total enthalpy in the range of 300 to 1200° F exists below 900° F. Thus the salt could be used at temperatures below 1200° F without incurring a severe penalty in usable enthalpy. It is also significant that more than half of the thermal energy within this range is in fusion at 800° F. The heat capacity of the mixture was measured by means of an ice calorimeter.

Thermal Conductivity. Figure 8 shows the results of studies of salt thermal conductivity in the solid state. The conductivity of the eutectic is slightly lower than those of LiF, LiOH, or LiH. However, the net thermal impedance of the LiF-LiOH mixture may be lower because the eutectic adheres to the heat-transfer surfaces without forming the voids that often occur in salts having high contraction during freezing.

CHANGE IN MELTING POINT

When the molten salt is maintained at high temperatures for extended periods, some of the salt decomposes. This decomposition is due to the breakdown of the LiOH to Li_2O and H_2O . The rate of this breakdown is greatly increased by corrosion and by the contamination of the salt by corrosion products. The breakdown occurs more rapidly in small containers because of the small ratio of salt volume to container surface area. Since the surface area varies as the square of the container radius, and the volume as the cube of the radius, the rate of corrosion and decomposition would be expected to decrease with large thermal storage units.

Decomposition of the salt changes the melting characteristics and raises the temperature at which the thermal energy is released upon solidifying. As decomposition proceeds, portions of the salt solidify at temperatures above 800° F. Figure 9 shows an example of the effects of this decomposition, which occurred when some of the salt was maintained at 1200° F for extended periods of time in a small container.

At lower temperatures, corrosion and salt breakdown are greatly reduced. Tests conducted for long periods at 900° F yielded little or no change or distortion of the melting point. This was the case even when 304 stainless steel was used as the container material.

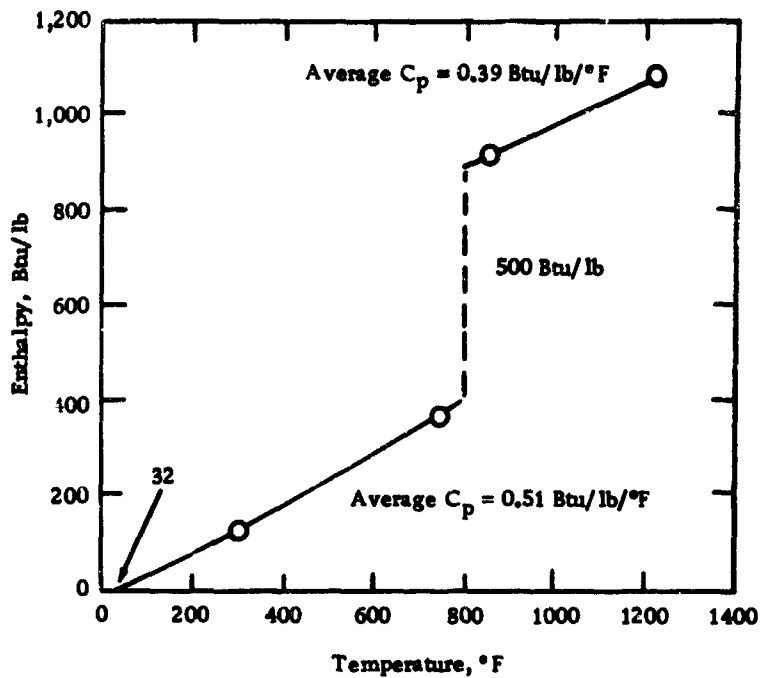


FIG. 7. Enthalpy Distribution for the LiF-LiOH Eutectic Mixture.

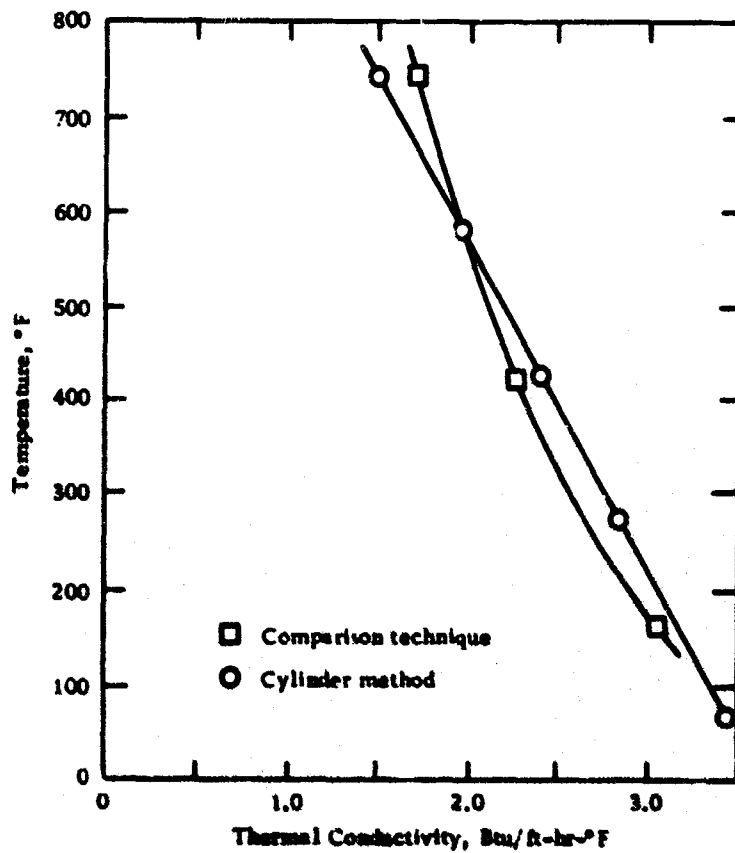


FIG. 8. Thermal Conductivity for the LiF-LiOH Eutectic Mixture.

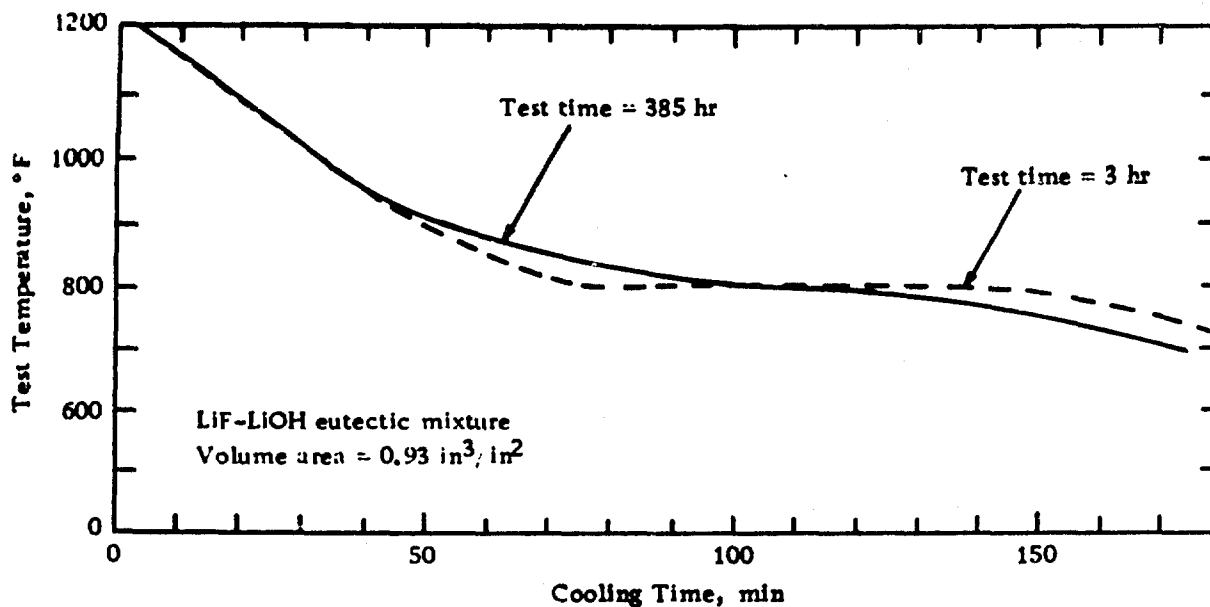
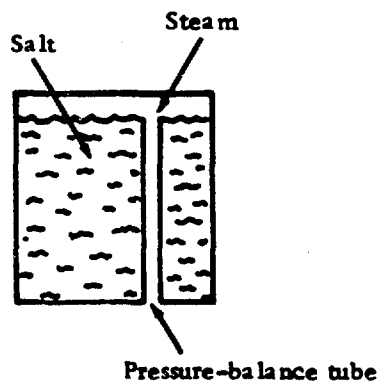


FIG. 9. Natural Cooling Curves Showing Distortion of the Energy Distribution Resulting From Extended Test Periods at 1200°F. Small test crucibles were used.

WATER SOLUBILITY IN THE EUTECTIC MIXTURE

The original system to which the salt was to be applied called for a pressure-balancing concept to eliminate the need for a heavy pressure vessel (see schematic). As the vehicle depth increases, more water is forced into the inlet tube and is turned to steam, until the pressure above the salt mass equals the ambient pressure of the ocean. When the vehicle moves to shallower depths, the excess high-pressure steam is expelled from the salt tank and condensed, so that the pressure balance is maintained at all times.



Although this pressure-balancing system offers a very simple approach to some buoyancy and structural problems, the solubility of water in the salts is great enough to impose problems with buoyancy control. Data from one experiment, conducted by Atomics International and pertaining to the solubility of water in the salt, are given in Table 2. In general, it was ascertained that water is more soluble in the molten salt than in the solid, but that the solubility decreases as the temperature is increased above the melting point.

TABLE 2. Solubility of Water in the Eutectic Mixture of LiF-LiOH

Phase	Temperature, °F	Pressure, psig	Solubility, g/100g of salt
Solid	800	1,000	2.4
Liquid	806	800	4.7
Liquid	1382	2,000	0.6

Several hours were required for the dissolution of the water to reach equilibrium solubility in this experiment. Upon freezing, however, the dissolved water was expelled rapidly from the melt. This rapid expulsion caused the salt to expand. When the melt was saturated with water immediately above the melting point, the expansion often exceeded the original volume of the melt.

This expansion phenomenon also occurred, but to a much lesser degree, when there was no steam or high pressure above the salts. The cause is presumed to be the release of dissolved gases in the salt. The expansion is dependent upon the rate of cooling. It was never witnessed during the tests in which the salt was allowed to cool slowly, but was easy to reproduce under rapid cooling conditions. Figure 10 shows a specimen that expanded when molten salt was poured into a cold iron mold. The molten salt had not been exposed to high pressures or a steam atmosphere.

CONCLUSIONS

The work conducted at the Naval Undersea Warfare Center has yielded data on a eutectic salt mixture with novel characteristics that should prove useful in both underwater and other thermal storage applications. The significant characteristics of the lithium fluoride-lithium hydroxide mixture are as follows:



FIG. 10. Expansion of the Salt Mixture Resulting From the Release of Dissolved Gases When the Salt Was Cooled.

1. Little or no volume change during fusion, and only about 7% volume change over the range from room temperature to 1200° F
2. Low melting point of 800° F
3. Good thermal capacity
4. Relative safety except for normal high-temperature hazards
5. No violent chemical reaction with water
6. Corrosivity with most container materials above 900° F, but with 800 Btu/lb of useful heat capacity available at temperatures below 900° F

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13. ABSTRACT <p>An analysis of various thermal storage materials indicated that a eutectic mixture of lithium hydroxide and lithium fluoride would provide relatively high heat capacity for a closed-cycle thermal propulsion system for deep-running underwater vehicles. In contrast with other high-heat-capacity lithium compounds, this eutectic mixture undergoes practically no expansion during fusion. It has a low melting point of 800°F. It does not react chemically with water, but does absorb small amounts of water if directly exposed to steam at high pressure. LiF-LiOH corrosion tests on samples of various metals, including 304 stainless steel and Inconel 600, indicated that corrosion may occur at random at temperatures above 900°F. From 900°F to 1200°F, Inconel 600 indicated the least corrosion of any high-strength material tested.</p>		

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