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STRUCTURAL MATERIALS IN LASL LIQUID SODIUM SYSTEMS*

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ABSTRACT

The major portion of this paper is devoted to a discussion of the design, fabrication, and post-operative tests and inspections of the 2000 kw Sodium Test Facility which was constructed and operated at the Los Alamos Scientific Laboratory. Of special interest are (1) post-operative inspection of heat exchanger tubing, (2) the investigation of a rupture in a thermocouple well, and (3) the investigation of the thermal stress fatigue cracking of a pipe tee and adjacent piping. Discussed briefly are (1) operating experience with 17-4 PH stainless steel in the LAMPRE sodium system and (2) the results of corrosion tests of tantalum in cold-trapped and hot-trapped sodium.

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INTRODUCTION

The LAMPRE* program at LASL has as its objective the development of high performance, fast reactor systems using molten plutonium alloys as fuels. This type of reactor requires a liquid metal coolant and in particular LASL has concentrated on the use of sodium. The material primarily used in these sodium systems has been the 300 series of stainless steels. However, for special application, 17-4 PH stainless steel and $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel have been used. Since tantalum and tantalum alloys are being used for fuel containment, the interaction between these materials and sodium is of importance. This paper will deal sequentially with the following topics:

1. Operating experience with stainless steel and $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel in the 2000 kw Sodium Test Facility.
2. Operating experience with 17-4 PH stainless steel in the LAMPRE sodium system.
3. Tantalum compatibility in sodium.

*Los Alamos Molten Plutonium Reactor Experiment.

OPERATING EXPERIENCE WITH STAINLESS STEEL AND

1 $\frac{1}{4}$ % Cr- $\frac{1}{2}$ % Mo ALLOY STEEL IN THE 2000 KW SODIUM TEST FACILITY

The 2000 kw Sodium Test Facility was constructed and operated to investigate the design integrity and operating characteristics of liquid sodium system components at temperatures up to 1100°F.

To familiarize the reader with the sodium test facility, a brief description of the sodium system components, operation, and fabrication and inspection techniques follows*.

Description of Sodium System

The test facility was designed to generate steam at 925°F and 900 psig from a sodium source at 1100°F. Design flow and temperature conditions for 100% load and a simplified flow diagram are shown in Fig. 1. The sodium system consisted of two loops, (1) the primary loop which included the gas-fired sodium heater, primary pump, and intermediate heat exchanger and (2) the secondary loop which included the steam generating equipment and secondary pump. Each loop also contained accessories such as flowmeters, valves, thermocouples, level indicators, and pressure transmitters. A single sodium purification system and a single dump and fill tank served both loops. The primary loop was fitted with remotely operated valves in the piping to the gas-fired heater and its bypass piping to permit thermal cycling of the intermediate heat exchanger and steam generating equipment. The

*For a detailed description of the 2000 kw Sodium Test Facility see references 1 through 7.

secondary loop was fitted with remotely operated valves in the piping to the tempering heat exchanger and its bypass piping to permit control of the temperature of the sodium entering the evaporator. Shutoff valves were installed only where absolutely necessary in drain and fill piping and in sodium purification system piping.

The intermediate heat exchanger was a vertical U-tube unit constructed of Type 316 stainless steel. The unit was designed for primary sodium flow through the shell and secondary sodium flow through the tubes. A thermal barrier of helium gas was provided in each leg between the shell sodium and the tube sheet. Figure 2 shows this unit during construction.

The steam generating equipment consisted of three separate heat exchange sections (1) an evaporator, (2) a sodium-to-sodium tempering heat exchanger, and (3) a superheater. The shells of all three heat exchangers were connected in series to form one continuous U-bend unit as shown in Fig. 3. Each heat exchange section was of the vertical U-tube type with a long horizontal run furnishing the heat transfer surface. Sodium flow was in the shells of all heat exchangers, with water and steam in the tubes of the evaporator and superheater, and sodium in the tubes of the tempering heat exchanger. A thermal barrier of helium gas was provided in each vertical leg between the shell-side sodium and the tube sheet. The superheater was Type 316 stainless steel whereas the tempering heat exchanger and evaporator were $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel.

The function of the tempering heat exchanger was to reduce temperature differences and thermal stresses in the evaporator. As seen in Fig. 1, the sodium outlet temperature from the superheater was 938°F while the saturated steam outlet temperature from the evaporator was 535°F. If the 938°F sodium had been introduced directly into the evaporator, excessive thermal stresses would have existed in the walls of the evaporator tubes. An economizer heat exchanger, called the tempering heat exchanger, was installed between the superheater and evaporator thereby reducing temperature differences and thermal stresses.

The two liquid sodium pumps were vertical centrifugal type installed in suction tanks. The shaft seals were located above a helium-filled expansion volume in each suction tank and were not in direct contact with sodium. Pumps and tanks were constructed of Type 304 stainless steel.

Sodium purification was accomplished by means of a conventional cold trap and plugging indicator system which could be valved for operation with either the primary or secondary system. All material was Type 304 stainless steel.

Sodium Purity

Extreme care was taken during construction to keep the piping and equipment as clean as possible. Before being filled with sodium the system was heated to approximately 500°F and maintained under a vacuum continuously for about nine days. Sodium was then circulated in each loop and a small amount bypassed to the sodium purification system until the plugging temperature dropped to 220°F

which corresponds to an oxygen content of less than 10 ppm. The initial sodium cleanup operation required one week for each loop.

During operation the sodium purification system continuously monitored the secondary loop to detect water-to-sodium leaks in the evaporator or superheater. The plugging temperatures were checked periodically in both loops and remained at 220°F indicating freedom from water-to-sodium reactions.

Thermal Transients

The facility was operated at constant load with a small number of thermal transients consisting of approximately 40 slow transients due to startup and shutdown, approximately 1200 slow transients due to change in load and approximately 40 severe transients due to accidental electric power failures and programmed tests.

Basis for Material Selections

Structural materials were selected on the basis of compatibility with liquid sodium and suitability for the temperatures involved. A ferritic alloy steel, rather than austenitic stainless steel, was selected for the tempering heat exchanger and evaporator. The relatively higher thermal conductivity and lower thermal expansion coefficient of the ferritic alloys tend to reduce thermal stresses. Except for low carbon stainless steels design working stresses were not permitted to exceed the allowable stresses set forth in the ASME Code for Unfired Pressure Vessels and the ASA Code for Pressure Piping. Table I contains a list of sodium system components, their design temperatures, and the structural materials selected.

Fabrication and Quality Control

The sodium system was of all welded construction except for flanged connections between the sodium pumps and suction tanks, rupture disc flanges on the heat exchangers, and a flanged manhole on the dump and fill tank. Transition welds between stainless steel and $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel were made using Inco Rod "A" for the filler material. No preheat was used and parts were allowed to cool to room temperature in still air after welding. Stainless steel welds and transition welds were relatively easy to make and were of consistently high quality; whereas, $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel welds were difficult to make and of relatively poor quality.

A fully radiographable, crevice-free, tube-to-tube sheet joint design¹ was used for the intermediate heat exchanger, superheater, and tempering heat exchanger. The welds were made with an internal welding gun utilizing the TIG process. A rolled and welded tube-to-tube sheet joint design was used for the evaporator.

A variety of nondestructive inspection techniques was employed because of the necessity of obtaining a sodium system of high integrity and of maintaining an impervious barrier between water and sodium in the superheater and evaporator. Table II is a summary of the inspection techniques used during fabrication of the various components.

Post-operative Tests and Inspections

Approximately 18 months after initial startup, operation of the sodium test facility was terminated and the equipment was dismantled for inspection. Total

operating times logged on major items of equipment are shown in Table III.

Post-operative tests and inspections have, with two exceptions, revealed little damage. In particular, visual and metallographic examination of transition welds between stainless steel and $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel revealed no damage. Post-operative tests and inspections to be discussed herein are (1) nondestructive inspection of tube-to-tube sheet welds in the heat exchangers, (2) investigation of a rupture in a thermocouple well, (3) nondestructive inspection, physical testing, and metallographic examination of heat exchanger tubing, and (4) investigation of cracks in a pipe tee and adjacent piping.

Nondestructive Inspection of Tube-to-Tube Sheet Welds

Post-operative dye penetrant inspection of all tube-to-tube sheet welds disclosed one crack-like indication in a superheater weld. Radiographic inspection revealed no detectable changes in any of the superheater welds. In the tempering heat exchanger, however, a few additional cavities or pits, and some crack-like indications with axes oriented parallel to the axis of the tube were observed in two welds. None of the defects observed compromised the integrity of the system and do not imply that a failure was imminent.

Investigation of Rupture in Thermocouple Well

One of three thermocouple wells installed between the superheater and tempering heat exchanger, operating in sodium at about 900° F, was found to be severely cracked on both the inside and outside diameters, and contained a complete rupture. These wells were fabricated from 5/16-in. o.d. x 0.035-in. wall, Type 316 stainless steel tubing with the ends closed by spinning and welding. The

observed cracks were intergranular and appeared to be the result of carbide precipitation in the grain boundaries. Figure 4 is a photograph of the outside of the thermocouple well showing the rupture. Figure 5 is a metallographic section through the rupture area showing many cracks propagating from the inside diameter. Sodium leaking into the well reacted with the ceramic insulating material on the thermocouple leads and plugged the well completely, thus preventing leakage of sodium to atmosphere and preserving the integrity of the sodium system.

Inspection of Heat Exchanger Tubes

Nondestructive Inspection. After disassembly of the heat exchangers, all tubes were subjected to dye penetrant inspection. Tubes from the evaporator, tempering heat exchanger, and superheater which yielded penetrant indications were also subjected to radiographic and ultrasonic inspections. Results of non-destructive inspections both after and before operation are summarized in Table IV. Three indications which had been observed before operation had increased in size, and 19 indications which were not apparent before operation were found. Inspection of Table IV indicates that perhaps dye penetrant inspection was the most sensitive of the methods employed for finding outside surface defects.

Physical Tests. Physical tests were conducted on specimens of heat exchanger tubing to determine if significant changes in mechanical properties occurred during operation. Results of these tests are shown in Table V. The Type 316 stainless steel tubing increased slightly in hardness and tensile strength

and decreased in ductility, while the $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo showed just the opposite effect.

Metallographic Examination of Intermediate Heat Exchanger Tubes (Type 316 Stainless Steel). Transverse metallographic specimens were cut from tubes I4 and I10. The microstructures of the hot ends of both tubes showed considerable carbide precipitation in the grain boundaries while the cold ends appeared normal and similar to the as-received tubing. Figure 6 shows the appearance of the sensitized material and Fig. 7 shows the outside surfaces of as-received tubing and the hot end of tube I10. There was a roughened appearance on the outside surfaces at the hot ends due to minor pitting corrosion. The inside surfaces had a similar appearance but did not differ significantly from that of as-received tubing, making it difficult to determine which irregularities were caused by corrosion and which were caused by the tube manufacturing process. There was no evidence of scale deposits, sigma formation, or intergranular penetration in any of the tubes.

Table VI shows average metal operating temperatures and the results of microhardness tests. The as-received hardness was 165 to 170 DPH; after service the hot ends were 188 to 192 DPH and the cold ends were 176 to 181 DPH. These increases in hardness and the slight increases in tensile strengths reported in Table V are believed to be the result of precipitation hardening during service.

Metallographic Examination of Superheater Tubes (Type 316 Stainless Steel). Transverse specimens were cut from the hot and cold ends of tubes S4, S9, and S10. All of the hot end specimens contained carbides precipitated in the grain boundaries.

The cold end specimens appeared to contain less grain boundary carbide precipitation than the hot ends, and in tube S10 the cold end showed a slight amount of precipitation in the twin boundaries only. Typical appearance of microstructures with grain boundary carbide precipitation is shown in Fig. 8. The amount of precipitation appeared to be greater near the inside and outside diameters than at the middle of the wall thickness. There was no evidence of sigma formation or intergranular corrosion, and the outside surfaces were smooth without pitting. Scale deposits were found on the inside surfaces of all specimens. This scale was continuous around the inside surface and probably originated in the feedwater. Thicknesses of scale deposits are shown in Table VII. With the exception of tube S9 the deposits were thicker in the hot end than in the cold end. Figure 9 shows typical scale deposits which do not appear to follow intergranular paths.

Microhardness measurements reported in Table VI and the slight increases in tensile strengths reported in Table V indicate precipitation hardening during service. The as-received tubing had a hardness of 170 DPH, the cold ends increased slightly to 173 to 176 DPH (specimen S10-3 decreased to 167), and the hot ends increased to 181 to 189 DPH. The hardness and tensile strength data coincide with the appearance of the microstructures, i.e., the harder, hot end specimens appeared to have precipitated more carbides than the softer, cold end specimens. Although changes in mechanical properties of the Type 316 stainless steel intermediate heat exchanger and superheater tubes are associated with increased amounts of visible precipitates, it is believed that the higher hardness

values are the result of increased precipitation of unseen, coherent precipitates throughout the matrix and that the observed precipitates are merely symptomatic of the existence of a coherent precipitate. It is expected that prolonged service at these temperatures might eventually lead to overaging with associated softening and increased amounts of visible grain boundary precipitates.

Metallographic Examination of Evaporator Tubes ($1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo Alloy Steel).

Transverse specimens were cut from the hot and cold ends of tube E6 and from the hot and cold ends and middle of tubes E16 and E17. The tubing was purchased in the annealed condition and the only other heat treatment received prior to service was a stress relief, after fabrication, at 1350°F for $\frac{1}{2}$ hour with vacuum outside and helium inside the tubes. The as-received microstructure was that of a fully annealed ferritic steel with spheroidized carbides. The grain size was ASTM 8 at the surfaces graded to ASTM 10 at the middle of the wall. The only apparent change in microstructure after service was further agglomeration of the carbides and decarburization at the outside and inside surfaces. Figure 10 shows the microstructure before and after service.

Table VIII shows the results of microhardness measurements taken on the specimens at locations 50 microns from the outside and inside surfaces and at the middle of the wall. All specimens were softer than the as-received material; however, it is not known if this softening is attributable to the service environment or to the stress-relieving treatment since no specimens in the stress-relieved condition

are available. A softening due to decarburization occurred in all specimens with an average reduction in microhardness of 16 points at the outside surface and 15 points at the inside surface. Since the stress relieving was performed in vacuum and in helium, it is believed this decarburization is primarily a result of the service environment. There does not appear to be any significant variation with respect to decarburization of the hot and cold ends of the tubes. The depths of decarburization, measured visually, are shown in Table VIII; the average depth for all tubes being 0.009 in. both inside and outside.

A heavy scale deposit was found on the inside surfaces of all evaporator tubes, the thicknesses of which are shown in Table VII. The deposits ranged in thickness from 5 to 25 microns at the cold ends to 165 to 316 microns at the hot ends. All deposits consisted of a dense, continuous layer adjacent to the tube surface and over this a porous or spongy layer as shown in Fig. 11. Pits containing the scale were quite common and varied in depth from about 1 to 15 microns. The paths taken by the deeper pits appeared to be limited by grain boundaries as illustrated in Fig. 11.

Metallographic Examination of Tempering Heat Exchanger Tubes ($1\frac{1}{2}\%$ Cr- $\frac{1}{2}\%$ Mo Alloy Steel). The tempering heat exchanger was stress relieved after fabrication with a helium atmosphere both inside and outside the tubes. Apparently the atmosphere was not sufficiently pure, resulting in the formation of a considerable amount of scale on the tube surfaces. Therefore, no meaningful analysis can be made regarding decarburization or scale formation. Microhardness measurements

at the middle of the tube wall thicknesses were in the range of 141 to 156 (average 145) with no significant variations between the hot and cold ends. The microstructures appeared similar to those of the evaporator with similar agglomeration of carbides and grain size distribution. There was no evidence of either intergranular or pitting type corrosive attack.

Investigation of Cracks in Pipe Tee and Adjacent Piping⁸

Many cracks were discovered in a 2½-in. Type 304L stainless steel pipe tee and adjacent piping which were used to mix two streams of secondary-loop sodium. Figures 12 and 13 are photographs of inside surfaces showing the orientation of the damaged tee and adjacent piping and general locations of defects. The surfaces were treated with dye penetrant and developed to emphasize the cracks. The cold sodium stream was from the tube side of the tempering heat exchanger, the hot sodium stream was from the tempering heat exchanger bypass, and the mixed sodium outlet stream flowed to the tube side of the intermediate heat exchanger. Dye penetrant inspection revealed no damage to either the hot sodium or the cold sodium inlet piping. The damage appeared to be restricted to the tee and to that portion of the piping in which the hot and cold sodium streams were being mixed.

Two major cracks, symmetrically oriented and diametrically opposed in the upper and lower halves of the tee body, were found. The length of each major crack was about 1-3/4 in. and the major crack in the upper half was about 0.15-in. deep or approximately 75% of the total wall thickness. In addition numerous smaller cracks were found in the tee body. Figure 14 shows the transgranular nature of the

cracks. Figure 15 is a photomicrograph of a section cut from the tee showing a thin (0.0002-in. thick) case on the inside surface. The case hardness was 344 DPH compared with a bulk metal hardness of 109 DPH. The possibility of contribution to cracking by this case-hardened surface has not been determined.

The piping at the mixed flow outlet end of the tee was also severely cracked. As can be seen in Fig. 16 the cracks extended both circumferentially around the pipe and longitudinally along the pipe. The damaged region extended for a length of about 18 in. downstream from the side inlet centerline of the tee. The cracks were transgranular in nature and the depth of a typical crack was about $12\frac{1}{2}\%$ of the pipe wall thickness.

Average sodium stream temperatures varied with operating load conditions. From Table IX it can be seen that temperature differences between the hot and cold sodium streams during steady load operation varied from a maximum of 361°F to a minimum of 270°F. The tee and adjacent piping operated for a period of 9500 hours under the range of mixing temperature conditions shown in Table IX and for an additional 4000 hours with no temperature difference between hot and cold sodium streams. At no time during the entire operating period was the sodium temperature lowered below 450°F, nor was it ever above 875°F.

An analysis of operating conditions and physical arrangement indicates that pressure stresses, hanger load stresses, and pipe thermal expansion stresses were much lower than those which might reasonably have caused failure. The nature of the major cracks in the tee suggests that the damage was caused by strains

induced by temperature gradients along the tee wall. Such strains might have arisen from (1) a relatively steady temperature gradient along the tee wall caused by the temperature difference between the hot and cold sodium inlet streams and (2) localized temperature gradients on the inside metal surface resulting from rapid fluctuations in temperatures caused by the mixing of the hot and cold sodium inlet streams.

Stresses induced by relatively steady state temperature differences should have been self-relieving as a result of metal yield; and no permanent damage is believed to have resulted from the relatively small number of such cycles. An analytical determination of the rate and magnitude of temperature transients within the metal as a result of mixing action would be most difficult and was not attempted. Although the magnitude of the cyclic variation in localized metal temperatures may have been relatively small, the rate of temperature change was quite high and the number of cycles conceivably astronomical. It is believed that the many metal flexures caused by this mixing action was the predominant factor in initiating and propagating the observed damage in the tee. The cracks found in the mixing pipe were caused solely by cyclic temperature conditions resulting from the mixing action since this area could not have been seriously stressed by temperature gradients in the inlet piping.

Conclusions and Recommendations

Operating experience with the sodium test facility was, in general, satisfactory. Since post-operative tests and inspections have, with two exceptions,

revealed little damage to structural materials, it is believed that mechanical design and selection of materials was generally satisfactory. The following conclusions and recommendations are presented:

1. Since visual and metallographic examination of transition welds between stainless steel and $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel revealed no damage, these transition welds are believed satisfactory for operation in liquid sodium at 500-950°F.
2. The fully radiographable, crevice-free, tube-to-tube sheet joint design appears to be satisfactory for sodium-to-sodium heat exchangers and for sodium-heated steam generating equipment fabricated from Type 316 stainless steel. Its use with equipment fabricated from $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel is not recommended because of poor weld quality.
3. It appears that the failure of the Type 316 stainless steel thermocouple well was caused by intergranular carbide precipitation. However, other thermocouple wells in the system including two which were installed in close proximity to the ruptured unit were found to be undamaged. It is suspected that cracking may have been accelerated by an undetected flaw or by high residual stresses or perhaps a combination of both.
4. The tubing employed in the sodium-heated steam generating equipment initially contained a number of 3 to 5 mil defects as shown by

ultrasonic inspection. Following operation a reinspection of the tubing showed that these defects had increased in number and size. However, both the tubing quality and the nondestructive testing techniques proved to be satisfactory for this particular application.

5. Although metallographic examination revealed grain boundary carbide precipitation and physical testing indicated slight increases in hardness and tensile strength in Type 316 stainless steel heat exchanger tube specimens, these changes did not significantly affect the structural integrity of the equipment.
6. The $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel heat exchanger tube specimens were decarburized to a depth of about 0.009 in. and decreased in hardness and tensile strength. Again, these changes did not significantly affect the structural integrity of the equipment. It might be advisable, however, to reduce the allowable design stresses for this material under similar operating conditions to compensate for the reduced tensile strength resulting from decarburization.
7. The cracks in the Type 304L stainless steel mixing tee and adjacent piping are believed to be the result of thermal stress fatigue caused by cyclic temperature conditions resulting from the mixing of the hot and cold sodium streams. When it is necessary to mix sodium streams of different temperatures, it is recommended that a mixing chamber with a thermal barrier be provided to protect the tee or containment vessel from thermal cycles due to mixing.

OPERATING EXPERIENCE WITH 17-4 PH STAINLESS STEEL
IN THE LAMPRE SODIUM SYSTEM

Description

LAMPRE is the first reactor built by LASL in its program to develop plutonium fuels for fast breeder reactor applications.⁹ The reactor is liquid sodium cooled, molten plutonium fueled, and designed principally for irradiation of fuel and container materials. A fuel capsule assembly is shown in Fig. 17. The fuel capsule at the lower end is fabricated from tantalum and filled with plutonium fuel except for a gas volume at the upper end. The closure plug is also fabricated from tantalum and welded to the capsule. A threaded Type 304 stainless steel adapter is attached to the tantalum closure plug with a Type 304 stainless steel dowel pin. The capsule handle which screws to the threaded adapter is fabricated from 17-4 PH stainless steel except for the top 2-in. section which is Type 304 stainless steel. Fuel capsules are inserted into and withdrawn from the core and maintained in the desired radial core configuration by these handles. The 17-4 PH portion of the fuel capsule assembly is approximately 9-ft long and about $\frac{1}{2}$ -in. diameter. Dummy fuel capsules of 17-4 PH stainless steel were fabricated to facilitate the initial stacking of fuel capsules in the core. Reflector pins were also fabricated from 17-4 PH stainless steel and are similar to the dummy fuel capsules except they are slightly larger in diameter.

Type 17-4 PH stainless steel was selected because it was believed it would be less subject to galling in sliding contact on the hexagonal surfaces and at the threaded connections. All material was purchased in condition A (solution treated at 1875-1925°F), the parts were then machined and hardened to condition H-950 in a vacuum furnace.

Operating Experience

The upper 3 ft of fuel capsule and reflector pin handles operate in a helium cover gas atmosphere. The remainder is exposed to flowing, zirconium-hot-trapped sodium. Sodium system shakedown operation at 575-930°F with dummy capsules in the core required about three months. Mechanical properties of 17-4 PH stainless steel removed from LAMPRE after the shakedown and before criticality are compared with mechanical properties of unexposed material in Table X. There was no change in hardness; however, there was a significant decrease in impact strength which is probably attributable to an overaged condition as shown in the photomicrographs in Fig. 18.

A copper-brown color has been observed on some handles beginning at a point about 3 ft from the top extending down about 12 in. This is near the sodium-helium interface. A chemical spot test reveals a high concentration of copper on the surface. A similar test on new material does not give this indication. The only known source of copper in the LAMPRE system is the 3½% contained in the 17-4 PH stainless steel. The copper-brown color has also been observed on

17-4 PH specimens exposed to flowing sodium in a test loop. At this time it is assumed that the presence of copper is due to diffusion from the 17-4 PH stainless steel.

Conclusions

It is believed that the overaging of 17-4 PH stainless steel in the LAMPRE system is entirely due to temperature and that the liquid sodium environment has no effect. The use of 17-4 PH stainless steel under similar temperature conditions is not recommended.

TANTALUM COMPATIBILITY IN SODIUM

Tantalum compatibility in sodium has been studied at the Los Alamos Scientific Laboratory¹⁰ and at Battelle Memorial Institute¹¹ as a part of the LAMPRE program. The investigations at LASL were of two types, (1) tantalum corrosion rates in tantalum-hot-trapped sodium and in cold-trapped sodium and (2) tantalum and zirconium corrosion behavior in cold-trapped sodium. The work at Battelle was an investigation of the corrosion of commercial tantalum in high purity flowing sodium.

Figure 19 shows the corrosion rates of tantalum in cold-trapped sodium and in tantalum-hot-trapped sodium.¹⁰ Weight losses for tantalum in cold-trapped sodium begin at about 1075°F and increase to an equivalent of about 8 mils/yr at 1220°F. Weight losses for tantalum in hot-trapped sodium begin at about 970°F and increase to an equivalent of about 0.5 mil/yr at 1220°F. Absolute values of oxygen content of the sodium were not obtained in any of these tests because the oxygen concentrations were less than those which could be detected by the existing analytical methods. Figure 20 shows the results of tests made to compare the corrosion behavior of tantalum and zirconium in cold-trapped sodium. The results indicate that zirconium absorbs oxygen forming an adherent film and gains weight while tantalum absorbs oxygen and forms oxides which spall off with a resultant loss in weight.

In hot-trapped sodium systems which were operated for periods up to fifty days, weight losses sustained by tantalum specimens in 1200°F sodium were equivalent to about 0.1 mil/yr.¹¹ Weight losses sustained by tantalum specimens in 1200°F cold-trapped sodium were equivalent to about 3 mils/yr. The oxygen content of the cold-trapped sodium was about 40 ppm and that of the hot-trapped sodium was estimated to be much less than 10 ppm. For the most part examination of metallographic specimens revealed no deleterious corrosion effects. However, one group of tubing specimens made from arc cast tantalum that were operated in sodium with an oxygen content of about 80 ppm suffered severe intergranular attack. This group of specimens also revealed a peculiar microstructural platelet feature which has also been observed in the welds of LAMPRE fuel capsules examined after irradiation. In LAMPRE fuel capsules the platelets have been found only in the weld zone where the closure plug is welded to the fuel capsule, and they generally occur around the periphery intersecting the outside surface of the weld as illustrated in Fig. 21. The chemical composition and mechanism of formation of these platelets has not yet been determined.

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TABLES

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- VII. Scale Thickness.
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from LAMPRE Sodium System

TABLE I
DESIGN TEMPERATURES AND STRUCTURAL MATERIALS

<u>Component</u>	<u>Design Temperature, °F</u>	<u>Structural Material</u>
Pipe and fittings	1100	Type 304L stainless steel
Valves	1100	Type 316 stainless steel
Tube bundle in gas-fired sodium heater	1300	Type 316 stainless steel
Intermediate heat exchanger	1100	Type 316 stainless steel
Superheater	1000	Type 316 stainless steel
Tempering heat exchanger	1000	1 $\frac{1}{4}$ % Cr- $\frac{1}{2}$ % Mo alloy steel
Evaporator	800	1 $\frac{1}{4}$ % Cr- $\frac{1}{2}$ % Mo alloy steel
Pumps and suction tanks	900	Type 304 stainless steel
Sodium purification equipment	1000	Type 304 stainless steel
Dump and fill tank	500	Carbon steel
Flowmeter tubes	1100	Type 304L stainless steel
Pressure transmitters	1100	Type 316 stainless steel
Thermocouple wells	1100	Type 316 stainless steel

TABLE II

NONDESTRUCTIVE INSPECTION TECHNIQUES EMPLOYED

<u>Component</u>	<u>Dye Penetrant</u>	<u>Ultrasonic</u>	<u>Radiographic</u>	<u>Hydrostatic</u>	<u>Helium Mass Spectrometer Leak Test</u>
1. Gas-fired sodium heater					
Tubes	X	X			
Headers	X	X			
Welds	X		X		
Completed unit				X	X
2. Heat exchangers					
Forgings		X			
Tubes	X	X	X	X	
Welds in prefabricated shells	X		X		
Tube-to-tube sheet welds					
Intermediate heat exchanger	X		X		X
Superheater	X		X	X	X
Evaporator	X				X
Tempering heat exchanger	X		X		X
Closure welds					
Intermediate heat exchanger	X		X		X
Superheater	X				X
Evaporator	X				X
Tempering heat exchanger	X		X		X
3. Valves					X
4. Pump suction tanks	X		X		X
5. Sodium purification equipment	X				X
6. Dump and fill tank				X	X
7. Piping system welds	X				X
8. Thermocouple wells	X		X		X

TABLE III

TOTAL OPERATING TIME

<u>Component</u>	<u>Hours</u>
Steam generating equipment	9, 509
Intermediate heat exchanger	11, 920
Primary sodium pump	11, 920
Secondary sodium pump	13, 474
Gas-fired sodium heater	10, 650
Sodium purification system	13, 662

TABLE IV

RESULTS OF NONDESTRUCTIVE INSPECTION OF HEAT EXCHANGER TUBES

<u>Heat Exchanger</u>	<u>Specimen</u>	<u>After Operation</u>			<u>Before Operation</u>		
		<u>Radiographic</u>	<u>Ultrasonic</u>	<u>Dye Penetrant</u>	<u>Radiographic</u>	<u>Ultrasonic</u>	<u>Dye Penetrant</u>
Evaporator	A4A	Negative	Negative	Linear	Negative	Negative	Negative
Tempering heat exchanger	A12A	Indication	0.003" & 0.005"	Nicks	"	"	"
Superheater	A5A-1	Negative	0.004"	Scab	"	"	Indication
"	A5A-2	"	Negative	Linear	"	"	Negative
"	A5A-3	"	0.003"	Linear	"	"	"
"	A6	"	0.008"	Linear	"	0.002"	"
"	A6B	"	0.005"	Scab	"	Negative	Indication
"	A6D	"	0.006"	Linear	"	"	Negative
Inermediate heat exchanger	1-1	Not inspected	Not inspected	Linear	"	"	"
"	1-2	"	"	Linear	"	"	"
"	3-1	"	"	Spot	"	"	"
"	3-2	"	"	Spot	"	"	"
"	3-3	"	"	Spot	"	"	"
"	4-1	"	"	Spot	"	"	"
"	4-2	"	"	Scab	"	"	"
"	7-1	"	"	Scab	"	"	"
"	9-1	"	"	Spot	"	"	"
"	9-2	"	"	Spot	"	"	"
"	11-1	"	"	Weld spatter	"	"	"
"	11-2	"	"	Linear	"	"	"
"	15-1	"	"	Spot	"	"	"
"	19-1	"	"	Spot	"	"	"

TABLE V

MECHANICAL PROPERTIES OF HEAT EXCHANGER TUBES

	Average Metal Operating Temperature ° F	<u>Elongation</u>		Outside Surface Hardness DPH (5 kg load)	<u>*Tensile Strength, psi</u>	
		Gage length % in.	Yield		Ultimate	
<u>Intermediate heat exchanger (Type 316 SS)</u>						
As received (a)	---	52	3.5	189	48,000	90,200
After use (a)	1000	44	3.5	207	49,600	95,100
After use (a)	760	45	3.5	201	47,600	94,500
<u>Superheater (Type 316 SS)</u>						
As received (b)	---	49	5.2	176	51,400	90,100
After use (c)	960	42	5.2	195	51,900	92,400
After use (c)	870	41	5.2	208	50,100	88,200
<u>Tempering heat exchanger (1$\frac{1}{4}$% Cr-$\frac{1}{2}$% Mo)</u>						
As received (b)	---	26	3.7	184	57,400	73,600
After use (d)	840	35	3.7	130	35,600	66,000
After use (d)	580	34	3.7	128	39,200	66,400
<u>Evaporator (1$\frac{1}{4}$% Cr-$\frac{1}{2}$% Mo)</u>						
As received (b)	---	22	5.1	167	47,900	72,500
After use (d)	680	30	5.1	138	37,200	62,200
After use (d)	540	31	5.1	130	40,300	61,600
After use (d)	500	30	5.1	131	42,200	64,400

(a) Average of 3 samples

(b) Average of 6 samples

(c) One sample

(d) Average of 2 samples

*Strain rate: 0.020 in./min to yield, 0.10 in./min to ultimate.

TABLE VI

MICROHARDNESS OF TYPE 316 STAINLESS STEEL

HEAT EXCHANGER TUBES

<u>Specimen</u>	<u>Average Metal Operating Temperature, ° F</u>	<u>Microhardness DPH (100 g load)</u>
<u>Intermediate heat exchanger</u>		
I10	As received	170
I26	As received	165
I10-1	1000	192
I10-6	760	176
I4-1	1000	188
I4-6	760	181
<u>Superheater</u>		
S49	As received	170
S4-1	960	182
S4-2	870	173
S9-1	960	181
S9-2	870	176
S10-1	960	189
S10-3	870	167

TABLE VII

SCALE THICKNESS

<u>Specimen</u>	<u>Average Metal Operating Temperature, ° F</u>	<u>Thickness, microns</u>
<u>Superheater</u>		
S4-1	960	10
S4-2	870	2
S9-1	960	8
S9-2	870	20
S10-1	960	60
S10-3	870	7
<u>Evaporator</u>		
E6-1	680	234
E6-3	500	5
E16-1	680	165
E16-2	540	75
E16-3	500	50
E17-1	680	315
E17-2	540	50
E17-3	500	25

TABLE VIII

MICROHARDNESS AND DECARBURIZATION OF EVAPORATOR TUBES

Specimen	Average Metal Operating Temperature, ° F	Microhardness*			Decarburization**	
		DPH (100 g load)			Depth, in.	
		<u>o.d.</u>	<u>Middle</u>	<u>i.d.</u>	<u>o.d.</u>	<u>i.d.</u>
E15	As received	161	156	159	0.000	0.000
E6-1	680	128	144	128	0.011	0.012
E6-3	500	132	145	126	0.008	0.010
E16-1	680	128	146	132	0.008	0.005
E16-2	540	128	147	132	0.007	0.008
E16-3	500	133	151	130	0.008	0.007
E17-1	680	122	132	124	0.009	0.006
E17-2	540	110	133	119	0.007	0.009
E17-3	500	128	139	128	0.012	0.010

*Measurements made on transverse metallographic specimens, o.d. and i.d. measurements were 50 microns from outside and inside surfaces, middle measurements were halfway between outside and inside surfaces.

**Determined visually.

TABLE IX

TYPICAL OPERATING SODIUM TEMPERATURES IN MIXING TEE

<u>% Rated Steam Load</u>	<u>Mixed Sodium Flow lb/hr</u>	<u>Sodium Temperatures, ° F</u>			
		<u>Cold In</u>	<u>Hot In</u>	<u>Mixed</u>	<u>Hot-Cold ΔT</u>
100	90,500	511	781	731	270
100	82,500	521	832	699	311
75	72,500	507	798	734	291
75	62,500	511	856	687	345
50	54,700	501	809	743	308
50	41,700	514	875	686	361

TABLE X

ROOM TEMPERATURE PHYSICAL PROPERTIES OF

17-4 PH STAINLESS STEEL FROM LAMPRE SODIUM SYSTEM

<u>Item</u>	<u>Impact Strength*</u> <u>ft-lb</u>	<u>Tensile Strength</u> <u>psi</u>	<u>Elongation</u> <u>in 2 in., %</u>	<u>Hardness</u>
Reflector pins, condition H-950	12.0 12.0			45 R _C
Reflector pins, originally condition H-950 and exposed to 575-930° F sodium	3.5 2.0 6.5 2.5 2.0 2.5	190,000	7.6 6.2 3.6 5.6	45 R _C
Handle, condition H-950	19.0 8.5 8.0 3.5			45 R _C
Handbook values	30.	180,000	14	40 R _C

*Izod impact test using modified Type Z specimen 0.400-in. diameter.

FIGURES

1. Sodium flow diagram. 2000 kw Sodium Test Facility.
2. Intermediate heat exchanger during fabrication.
3. Steam generating equipment.
4. Rupture in thermocouple well.
5. Rupture area in thermocouple well. X90.
6. Intergranular carbide precipitation near inside surface of Type 316 stainless steel tube from hot end of intermediate heat exchanger. X1100. Specimen I10-1.
7. Comparison of outside surface of Type 316 stainless steel tube from hot end of intermediate heat exchanger with outside surface of as-received tube. (a) After service specimen I10-1. X500, (b) As-received specimen I10. X500.
8. Intergranular carbide precipitation near outside surface of Type 316 stainless steel tube from hot end of superheater. X1100. Specimen S9-1.
9. Scale deposit on inside of Type 316 stainless steel tube from hot end of superheater. X500. Specimen S10-1.
10. Comparison of microstructure of $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel tube from evaporator with microstructure of as-received tube. (a) After service specimen E16-3. X100, (b) As-received specimen E15. X100.

11. Scale deposits on inside of $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel tube from evaporator. (a) X100. Specimen E17-2, (b) Path taken by pit appears to be limited by grain boundaries, X500. Specimen E17-2.
12. Exploded view of pipe tee and adjacent piping. Surface treated with dye penetrant and developed to emphasize cracks.
13. Top quarter of tee and connecting section of mixing pipe. Surface treated with dye penetrant and developed to emphasize cracks.
14. Transgranular crack in top quarter of tee. X90.
15. Section from tee showing hardened case on inside surface. Case thickness ~ 0.0002 in. Case microhardness 344 DPH (15 g load). Bulk metal microhardness 109 DPH (15 g load). X500.
16. Surface cracks in mixed-flow piping downstream from tee. Surface treated with dye penetrant and developed to emphasize cracks.
17. LAMPRE fuel capsule assembly.
18. 17-4 PH stainless steel from LAMPRE reflector pin. (a) Heat treated to condition H-950 before service. X750, (b) Grain boundary precipitation after service in flowing hot-trapped sodium at 575° - 930° F about 3 months. X750.
19. Corrosion rates of tantalum in cold-trapped sodium and in tantalum hot-trapped sodium.
20. Corrosion of tantalum and zirconium after 144 hours in cold-trapped sodium.
21. Platelets in weld zone of irradiated LAMPRE fuel capsule. X334.

Fig. 1. Sodium flow diagram. 2000 kw Sodium Test Facility.

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Fig. 2. Intermediate heat exchanger during fabrication.

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MATERIAL :

SUPERHEATER - 316 SS

TEMPERING HEATER - 1 1/4 CR.-1/2 MO. STEEL

EVAPORATOR - 1 1/4 CR.- 1/2 MO. STEEL

Fig. 3. Steam generating equipment.

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Fig. 4. Rupture in thermocouple well.

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Fig. 5. Rupture area in thermocouple well. X90.

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Fig. 6. Intergranular carbide precipitation near inside surface of Type 316 stainless steel tube from hot end of intermediate heat exchanger. X1100. Specimen I10-1.

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(a) After service specimen I10-1. X500.

(b) As-received specimen I10. X500.

Fig. 7. Comparison of outside surface of Type 316 stainless steel tube from hot end of intermediate heat exchanger with outside surface of as-received tube.

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Fig. 8. Intergranular carbide precipitation near outside surface of Type 316 stainless steel tube from hot end of superheater. X1100. Specimen S9-1.

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Fig. 9. Scale deposit on inside of Type 316 stainless steel tube from hot end of superheater. X500. Specimen S10-1.

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(a) After service specimen E16-3. X100.

(b) As-received specimen E15. X100.

Fig. 10. Comparison of microstructure of $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel tube from evaporator with microstructure of as-received tube.

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(a) X100. Specimen E17-2.

(b) Path taken by pit appears to be limited by grain boundaries, X500. Specimen E17-2.

Fig. 11. Scale deposits on inside of $1\frac{1}{4}\%$ Cr- $\frac{1}{2}\%$ Mo alloy steel tube from evaporator.

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Fig. 12. Exploded view of pipe tee and adjacent piping. Surface treated with dye penetrant and developed to emphasize cracks.

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Fig. 13. Top quarter of tee & connecting section of mixing pipe. Surface treated with dye penetrant and developed to emphasize cracks.

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Fig. 14. Transgranular crack in top quarter of tee. X90.

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Fig. 15. Section from tee showing hardened case on inside surface. Case thickness ~ 0.0002 in. Case microhardness 344 DPH (15 g load). Bulk metal microhardness 109 DPH (15 g load). X500.

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Fig. 16. Surface cracks in mixed-flow piping downstream from tee. Surface treated with dye penetrant and developed to emphasize cracks.

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Fig. 17. LAMPRE fuel capsule assembly.

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(a) Heat treated to condition H-950 before service. X750.

(b) Grain boundary precipitation after service in flowing hot-trapped sodium at 575°-930° F about 3 months. X750.

Fig. 18. 17-4 PH stainless steel from LAMPRE reflector pin.

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Fig. 19. Corrosion rates of tantalum in cold-trapped sodium and in tantalum hot-trapped sodium.

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Fig. 20. Corrosion of tantalum and zirconium after 144 hours in cold-trapped sodium.

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Fig. 21. Platelets in weld zone of irradiated LAMPRE fuel capsule. X334.

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