QUARTERLY PROGRESS REPORT
October-November-December 1967

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U. S. Atomic Energy Commission
Chicago Operations Office
Argonne, Illinois

MASTER

EFFECT OF HIGH TEMPERATURE SODIUM
ON THE
MECHANICAL PROPERTIES
OF
CANDIDATE ALLOYS
FOR THE
LMFBR PROGRAM

January 1968

MSA RESEARCH CORPORATION
DIVISION OF MINE SAFETY APPLIANCES COMPANY
EVANS CITY, PENNSYLVANIA 16033

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January 1968

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THE EFFECT OF HIGH TEMPERATURE SODIUM ON THE MECHANICAL PROPERTIES
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SUMMARY

1. EFFECT OF HIGH TEMPERATURE SODIUM ON AUSTENITIC AND FERRITIC
STEELS

STATIC EXPOSURE RETORTS

The four static retorts are operational. The sodium chemistry for each unit is being characterized, and the cold traps are maintaining the oxygen levels at <10 ppm. Long-term exposures of austenitics, as well as both long-term and short-term exposures of ferritic alloys, will be initiated during the next report period.

LOOP 1 - HIGH CARBON SODIUM ENVIRONMENTAL SYSTEM

The preliminary exposures are in progress. The exposures are necessary to resolve the actual operating conditions necessary to provide 15 mil specimens with a range of surface carbon contents from 0.3 to 0.5% in 100 hours. Evaluation of such specimens will determine the final carburizing conditions for the program's carbon excursion parameter.

LOOP 2 - HIGH OXYGEN SODIUM ENVIRONMENTAL SYSTEM

A recent decision was made to change the isothermal parameter of the high oxygen excursion to one containing a system ΔT . Therefore, the excursion is now defined as 500 hours of exposure at 1200 F in sodium containing 50 ppm oxygen as regulated by continuous operation of the cold trap which also imposed a ΔT on the system (the high temperature located at the specimen and the low temperature in the cold trap). All the scheduled units were made ready and, after one false start, the excursion was initiated and is in progress.

LOOP 3 - 'CLEAN' SODIUM MECHANICAL TEST SYSTEM

Preliminary operation is in progress while installation of the UNC oxygen meters and by-pass sampling system is completed. The actual installation work has been completed with the control and recording instrumentation still outstanding. Various flow studies with one test unit on stream have been made, but primary efforts have been on Loops 1 and 2 and the static units. Further flow studies, sodium chemistry characterization, and initial mechanical testing of specimens from Loops 1 and 2 are scheduled for the next report period.

2. EXTENSOMETER DEVELOPMENT PROGRAM

The success of operating two redesigned units in air at 1200-1300 F is encouraging. Longer exposure times at these high temperatures are still needed to verify the stability and durability of these units. Circuit weaknesses and errors have been spotlighted and studies are in progress in these areas.

3. DEVELOPMENT OF TECHNIQUES FOR MEASUREMENT OF IMPURITIES IN SODIUM

The surface-tension studies continue to be the only active work under this program. Surface tension measurements gave two different sets of values when the static system was operated at two different cold-trap temperature levels. Operating difficulties continue while attempting to define the actual relationship in more detail. At the present time, a contaminated surface layer is preventing accurate surface-level measurements. Steps have been taken to eliminate this condition before proceeding.

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INTRODUCTION

Operation of nuclear power plants cooled with sodium necessitates additional information regarding the effects of sodium and the normally anticipated contaminants on the mechanical properties of the structural materials. The basic program was originated to determine these effects on 316 ss and 2 1/4 Cr-1 Mo steel in dynamic sodium systems at 1200 F and 1100 F respectively, the anticipated operating temperature for each material. The mechanical property tests conducted in flowing sodium, air and helium to evaluate the environmental effects were creep, creep-rupture and fatigue. Two 316 ss loops (Loop 1 and Loop 2), designed and constructed under this contract, were used in carrying out the mechanical testing in liquid metal. Tensile and impact tests were run on pre-exposed and original material. The sodium contaminants studied were oxygen and carbon. The scope of testing was expanded to include 304 ss as one of the test materials.

Beginning in the fall of 1966, the program was directed into the study of the mechanical properties of candidate alloys for the LMFBR program. A third loop (Loop 3) and four static exposure units were added to the existing facilities. The test program consists of the exposure of 304 ss and 316 ss in sodium, with oxygen and carbon simulating contaminant excursions, and subsequent creep-rupture tests in clean sodium (<10 ppm O_2) with parameters of ΔT (50 to 150 F), velocities (15 to 30 ft/sec), temperature (1400 F maximum) and specimen thickness (15 to 65 mil). Long-time static exposures (10,000 to 20,000 hours), as well as the testing of ferritic-base, nickel-base, modified austenitic and special alloys, will be part of this program. Room temperature to 1400 F tensile tests on exposed materials will be used as a screening mechanism for choosing suitable alloys for further mechanical testing in sodium.

Full characterization of the sodium environments will be maintained using up-dated state-of-the-art methods and analyses.

Post-test evaluations of the effects of stress and corrosion will be made using metallographic and elemental analyses for oxygen, carbon, nitrogen, iron, nickel and chromium. In addition, microhardness measurements will be made to verify the above analyses. X-ray diffraction and microprobe analyses of corroded and deposited surfaces will be performed to help delineate chemistry and phase changes occurring during exposure.

Additional areas of investigation under this base program include a direct method of measuring creep rates in a high temperature liquid metal environment, the development of techniques for

measurement of impurities in sodium, and the analyses of two cold traps, -- one having been exposed to a high oxygen-sodium environment and the other to a high carbon-sodium environment.

PROGRAM PROGRESS

1. EFFECT OF HIGH TEMPERATURE SODIUM ON AUSTENITIC AND FERRITIC STEELS

MECHANICAL PROPERTY PROGRAM

Generally speaking, the present program is centered around the instream testing of both 'standard' and 'new' alloys after they have been exposed to high-temperature, contaminated-sodium environments. The testing will be the measure of rupture strength in 2000 to 3000 hours in 'clean' sodium (less than 10 ppm O₂) with parameters of velocity (15 to 30 ft/sec), material thickness (15 mil to 65 mil), ΔT (50 to 150 F), and temperature (maximum 1400 F). Exposures prior to this testing will include the simulation of oxygen and carbon excursions in circulating loops and long-time (10,000 to 20,000 hours) exposures in static retorts in oxygen-controlled sodium environments.

The general classes of structural materials which will be tested include the austenitics (304 and 316 ss), a series of modified or stabilized Cr-Mo ferritic alloys, selected vanadium and cobalt-based alloys as well as modified austenitic alloys. The specific alloys which have thus far been selected for testing are listed in Table 1, including their compositions as furnished by the various fabricators.

A condensed summary of the test program is presented in Table 2. The immediate program is concerned with the austenitics and Cr-Mo. The 304 and 316 ss alloys will be exposed (500 hours) to oxygen and carbon excursions followed by creep-rupture testing in 'clean' sodium for 2000 to 3000 hours under the various parameters previously listed. The results of these tests will then be compared to the mechanical property results from the previous tests with the same alloys. These included tests in air, helium, clean and contaminated sodium under isothermal and low velocity conditions. Specimens exposed to the contaminated sodium environments will also be tensile tested at temperatures of 70 to 1400 F.

A limited (cooperative) program may be initiated during the program to compare tube burst with uniaxial creep-rupture life and ductility.

Exposure of 304 and 316 ss to pure static sodium for 10,000, 20,000, and maybe even longer times will be carried out in sodium-filled retorts. The design of the retort permits both monitoring as well as control of the sodium purity throughout the exposures.

TABLE 1 - COMPOSITION OF TEST MATERIAL FOR MSAR-LMFBR STRUCTURAL MATERIAL PROGRAM

AUSTENITIC STEELS

<u>Type of Material</u>	<u>Supplier</u>	<u>Heat No.</u>	<u>Cr</u>	<u>Ni</u>	<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Mo</u>	<u>Other</u>
304	Allegheny Ludlum	322891	18.26	9.36	0.057	1.20	0.023	0.010	0.52	0.15	Cu = 0.16, Co = 0.090
316	Allegheny Ludlum	19295	17.64	12.72	0.048	1.68	0.019	0.011	0.56	2.85	Cu = 0.10

FERRITIC STEELS

<u>Type of Material</u>	<u>Supplier</u>	<u>Heat No.</u>	<u>Cr</u>	<u>Mo</u>	<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Other</u>
Mod. 2 1/4 Cr-1 Mo	U. S. S.	W8815	2.26	1.02	0.14	0.44	0.013	0.018	0.16	V = 0.62, Cb = 0.094, Al = 0.025
Stab. 2 1/4 Cr-1 Mo	B & W	--	2.25	1.01	0.10	0.45	0.023	0.011	0.49	Ti = 0.20, V = 0.20, Zr = 0.14, B = 0.005, Ni = 0.10, Co = 0.09
Rheinrohr 3HK5S	Thyssen Röhrenwerke		2.22	0.97	0.048	0.72	0.017	0.011	0.23	Ni = 0.61, Nb/Ta = 0.56
Croloy 5	B & W	47295	5.04	0.63	0.10	0.48	0.016	0.014	0.30	Ni = 0.23, Cu = 0.11
Croloy 5 Ti	B & W	10339	4.99	0.51	0.09	0.48	0.014	0.015	0.23	Ni = 0.24, Cu = 0.12, Ti = 0.45

TABLE 2 - SUMMARY OF MSAR-LMFBR STRUCTURAL MATERIAL TEST PROGRAM

Phase	Program	Facility
PHASE I - AUSTENITIC STEELS		
Task I	Oxygen excursions of 316 ss and 304 ss at 1200 F and 1400 F followed by creep-rupture tests in clean sodium.	High oxygen sodium (50 ppm) loop and clean sodium loop.
Task II	Carbon excursion of 316 ss and 304 ss at 1200 F and 1400 F followed by creep-rupture tests in clean sodium.	High carbon sodium loop and clean sodium loop.
Task III	Long-term (10,000 and 20,000 hrs) exposures of 316 ss and 304 ss in clean sodium followed by creep-rupture tests in clean sodium.	Static pots and clean sodium loop.
Task IV	Oxygen and carbon excursions on modified austenitic steel followed by creep-rupture tests in clean sodium.	High oxygen, high carbon and clean sodium loops.
Task V	Synergistic studies of carbon-oxygen exposures followed by creep-rupture tests in clean sodium.	High carbon, high oxygen and clean sodium loops.
PHASE II - FERRITIC STEELS		
Task I	Exposure in decarburizing environment followed by creep-rupture tests in clean sodium.	Static pots and clean sodium loop.
Task II	Long-time exposures (10,000 and 20,000 hrs) followed by creep-rupture tests in clean sodium.	Static pots and clean sodium loop.
PHASE III - OTHER CANDIDATE ALLOYS		
Task I	Creep-rupture tests in clean sodium.	Clean sodium loop.
Task II	Oxygen excursion followed by mechanical testing in clean sodium.	High oxygen loop and clean sodium loop.

Candidate ferritic steel alloys will be creep-rupture tested in clean sodium after exposures in clean sodium to induce decarburization and, in other cases, after long-time (10,000 to 20,000 hours) exposures. However, not all of the five exposed ferritic alloys will be creep-rupture tested. Tensile tests of temperatures of 70 to 1200 F will serve as a screening program so that only those alloys that show sufficient merit will progress to the creep-rupture tests in clean sodium. The exposure, screening and testing of the ferritic alloys and the exposure and testing of the austenitics will occupy the creep-rupture test units for FY68; however, preliminary testing and screening of exposed specimens of other alloys will be achieved by means of tensile tests.

Programs and testing of other alloys will evolve as additional specific candidate alloys are selected for testing.

PROGRAM STATUS - L. H. Kirschler

The primary effort during this report period was directed towards obtaining the proper operating conditions in Loop 1, Loop 2 and the static retorts and towards characterization of the sodium for each test condition.

For Loop 1, one of the operating conditions is to realize a carburizing activity that will produce a surface carbon content over the range of 0.3 to 0.5% for 15 mil specimens in approximately 100 hours.

Loop 2 requires an oxygen content of 50 ppm with exposure times of 500 hours. Since the last report period, a meeting was held at MSAR with representatives of RDT and the LMFBFR Program Office. At this meeting, a decision was made to change the isothermal system parameter during the high oxygen sodium excursion to a ΔT system. This will be done by continuous operation of the cold trap at the 50 ppm saturation temperature throughout the excursion period.

The static retort long-term exposures and the mechanical tests in Loop 3 are to be carried out in 'clean' sodium (less than 10 ppm O₂).

Additional efforts during this test period saw the installation of the UNC oxygen meter circuit, a bypass oxygen sampling circuit, and a special material exposure unit within Loop 3. All installations have been completed with the exception of the instrumentation for control of these additional circuits; therefore, none of the three special circuits has been put onstream as yet.

The operational status of each system and the results to date are discussed in the following sections.

Static Exposure Retorts - Long-Term Exposure Units

The installation was completed of the four static retorts for use in long-term exposures (10,000 hours and greater), as well as short times (1000 to 3000 hours), as defined under Phase I, Task III, and Phase II, Tasks I and II (see Table 2). The retorts were charged with a total of 154 pounds each of sodium and were brought to an anticipated operating temperature of 1200 F. Temperature profiles were made on each unit. The diffusional-type cold trap used to control the oxygen level indicated a minimum temperature of 490 F which was too high to anticipate an oxygen level of less than 10 ppm. Aluminum fins were press-fit onto each cold trap to provide additional surface area for cooling by either natural or forced circulation. At the present time, natural circulation alone has lowered the minimum operating temperature to <350 F on each of the units. At this time, duplicate sodium samples were taken with the following results:

Static Retort Oxygen Content, ppm

	<u>Retort #1</u>	<u>Retort #2</u>	<u>Retort #3</u>	<u>Retort #4</u>
Sample No. 1	<10	<10	<10	<10
Sample No. 2	<10	10	10	<10

These samples indicate that the operating temperatures of the cold traps are sufficiently low to maintain the test parameter for the long-term exposures. However, air movers have been purchased and forced-air cooling will be used for each unit. This should insure operation of <300 F operation under all ambient temperature changes.

Further samples are being taken to more fully characterize the sodium chemistry prior to initiating specimen exposures. These samples will be analyzed for carbon, carbon off-gases, hydrogen and metallic impurities. These determinations are presently in progress. Upon completion of these analyses, 304 ss monitoring tabs will be immersed into the sodium in each retort to characterize the carburizing activity of the sodium.

While this work is being completed, a helium gas system is being installed to permit simultaneous exposure of test specimens in a canned helium environment with those in the 'clean' sodium environment. These specimens will be used to differentiate thermal aging effects from corrosion effects.

It is anticipated that the sodium chemistry will be fully characterized and that the long-term exposure of 304 and 316 austenitic stainless steels and exposure of the candidate ferritic alloys for screening purposes will be initiated during the next report period.

Loop 1 - High Carbon Sodium Environmental System

This system will be used to simulate high carbon sodium excursions on test specimens prior to mechanical testing in the Loop 3 'clean' sodium, as defined in Phase I - Tasks II and IV. As discussed in the July-August-September 1967 Quarterly Report* and more recently, during the meeting at MSAR with representatives of RDT and the LMFBR Program Office, it was felt that a suggested 0.5% carbon/100-hour carbon limit would be too great. The decision was made to conduct a preliminary study whereby 5 mil carbon monitoring tabs, along with 15 mil tensile specimens, would be generated in Loop 1. These would provide specimens with a range of surface carbon content from 0.3 to 0.5% in 100 hours. The 15 mil specimens would then be tested and the results analyzed in an attempt to determine a suitable carbon level for conducting the full-scale tests.

The complexity of the sodium analyses in this stainless steel-sodium-carbon system was discussed at length in the last quarterly report also. Further discussion at the MSAR-RDT-LMFBR meeting led to the decision that the tests would be conducted using the plugging indicator and 304 ss monitoring tabs to follow the sodium chemistry as in previous tests. Meanwhile, additional analytical procedures would be employed to determine the discrepancies found between the cold trapping temperature, plugging temperature and analytical results. The work load in characterizing Loop 2 and the static retorts has limited these activities to date.

Early in this report period, operations of Loop 1 were such that there was a low flow (1/4 gpm) through the carbon bed and cold trapping was on a periodic-time basis. The 304 ss monitoring tabs during this period showed carbon increases to 14,000 to 18,000 ppm total carbon in ~165 hours. This greatly exceeded the carbon activity decided upon earlier. The mode of operation was then changed to the point where there was no flow through the carbon bed and cold trapping was continuous at 250 F. The time interval was also changed on the monitoring tabs to ~100 hours. Analyses of these tabs indicated a decrease of carbon activity to a point where a carbon increase to 4000 ppm in 100 hours was constant. On the basis of this data, the preliminary tests are scheduled to begin early in the next report period, utilizing the aforementioned mode of operation. Should the carbon activity again decrease during the test, as indicated by monitoring tabs, the flow through the carbon bed will be initiated to return the activity to the proper level. Further efforts to resolve the cold trap-plugging indicator-sodium analyses discrepancies are also scheduled in the following report period.

Midway through this report period, a temporary delay in operations occurred until a faulty circuit breaker for an auxiliary heater was replaced. All other operations were normal.

* MSAR 67-207

Loop 2 - High Oxygen Sodium Environmental System

Loop 2 is to be used to simulate high oxygen sodium excursions on test specimens prior to their mechanical testing in the Loop 3 'clean' sodium, as defined in Phase I - Tasks I and IV.

Initially, this excursion was to be carried out in a 1200 F isothermal system with an oxygen content of 50 ppm for a period of 500 hours. However, during the MSAR-RDT-LMFBR meeting, a decision was made to change from an isothermal system to one with a ΔT , -- all other parameters remaining the same. The addition of a ΔT to the system was to be accomplished by cold trapping continuously rather than on an intermittent basis and by making adjustments in the cold trap and bypass flow rates. Initial studies indicated that a ΔT of 50 F or more could be maintained between the test specimen temperature of 1200 F and the coldest spot in the main system. However, there exists a ΔT of ~ 675 F between the test-specimen temperature and the cold-trapping temperature. At the established flow rates, this cold-trap flow represents a significant portion ($\sim 30\%$) of total flow of the system flow which imposes a much greater driving force for corrosion and/or mass transfer.

In order to accurately define the above ΔT , it was necessary to drain the system and install an instream thermowell to measure the main stream ΔT . (After installation of this thermowell, studies verified that previous programs in this system were carried out with a maximum ΔT of 15 F in the main stream.)

Seven sodium samples were taken during this report period and were analyzed for oxygen. Plugging-indicator runs were made immediately after taking each sample. The results are as follows:

Loop 2 Sodium Chemistry

Analysis O ₂ ppm	Plugging Indicator	
	Saturation Temperature °F	Equivalent O ₂ ppm
47	510	56
43	510	56
49	510	56
47	495	44
43	490	43
50	500	48
51	500	48

Five of these samples fell within the 90% confidence limits set for the Loop 2 plugging meter calibration curve generated during the last report period. The two remaining results were just slightly below this limit.

With the system test parameters set and the sodium chemistry well defined, test specimens were placed into four of the

exposure units. Each unit contains a combination of 15 mil and 60 mil thick 304 and 316 ss tensile specimens totaling 30 specimens, along with 304 ss 5 mil monitoring tabs. A fifth unit was used to immerse a stainless steel can filled with a similar charge of specimens under a helium environment. These latter specimens will provide thermally aged specimens in order to separate thermal-aging effects from corrosion effects in the subsequent mechanical tests.

Initial efforts to put these units on-stream were unsuccessful when a sodium oxide-type plug in the expansion-tank cover-gas line caused a sodium level rise in several of the units. Two of the units leaked sodium around the top sealing flanges and, on one unit, sodium was forced into the cover-gas lines. At this point, all units were drained, the leaking flanges cleaned, seals replaced, and the cover-gas lines cleared. One unit had a plugged sodium valve which was also replaced.

At the present time, all repairs have been completed and excursions are in progress.

Loop 3 - 'Clean' Sodium Mechanical Test System

Loop 3 will be used to conduct all the mechanical tests in clean sodium, as defined under each of the tasks of Phases I, II and III.

While preliminary operations were being carried out, efforts continued on the installation of the UNC oxygen meter and bypass-sampling station circuits. This work is now complete with the exception of the temperature monitor-and-control systems for each. Two temperature instruments scheduled for delivery early in the following report period are to be installed. Wiring of thermocouples will then complete these two circuits.

The operations of Loop 3 were limited during this report period as a result of the workload imposed on both the engineering and laboratory personnel by the studies being conducted in Loop 1, Loop 2 and the static retorts. The main system has been circulating at 1200 F with the saturation temperature being maintained at 350 F or less. A single test unit was placed on-stream to check flow characteristics of the velocity flow blocks around the test specimens. Upon completion of this study, the test unit will be drained and the dismantling procedures performed. After evaluation of these procedures, several units will be placed on-stream and the flow characteristics and heat-load capabilities of the main system will be determined.

Sodium chemistry characterization is scheduled early in the next report period. The first exposed mechanical test specimens will be available in the early part of February. The initial mechanical tests should, therefore, be in progress during the next report period.

METALLURGY - R. Hiltz

The principal effort has been directed to the preparation of specimens from the various materials to be exposed during the program. Preparation of the majority of specimens was routine. The ferritic materials from B&W posed a major problem, however. They had been hot-rolled almost to finished size. The descaled material was badly marked with rolled-in defects. Due to the thinness of the material and the distortion resulting from the rolling operation, it was impossible to use mechanical machining procedures to clean up the specimen surface. Electrolytic machining was finally utilized but, even with this method, it was necessary in the long run to provide a final mechanical surface preparation to yield acceptable specimens. The amount of material removal required to reach this point was such that, in most cases, the final gauge thickness was of the order of 50 mils rather than the 60-65 mils originally intended.

In preparation for specimen testing, the new Instron Tensile Tester has been completely checked out. The high temperature furnace to be used in conjunction with it to test specimens in air has also been completely evaluated. A second furnace, -- a high temperature vacuum or inert atmosphere system -- has been undergoing extensive testing. Significant problems have occurred in achieving desired vacuum levels due to system leaks, principally in the bellows area. This furnace is still being worked upon.

In addition to the tensile unit, a second high-temperature creep rack has been installed and is ready for operation.

At present, all equipment, test units, and the like, are either operational or very close to that point. Specimens are almost complete, with the majority all prepared, cleaned, and ready for insertion into the loop. The ferritics and the modified stainless will be the last materials to be completed.

SODIUM CHEMISTRY - S. Rodgers

The analytical effort under this program includes characterization of the sodium charge of the three loops and four static retorts, plus the induction of surface tension under the Analytical Development Program. The plugging indicator of Loop 2 has been calibrated against chemical analysis for oxygen. These results have been compared with the Los Alamos oxygen solubility curve. Sampling and analysis schedules have been established for the loops and static pots. Preliminary data on the surface tension indicate a difference in relative surface-tension values depending on whether the cold trap is maintained hot or cold.

Loop 1 - Carbon Contaminated Sodium Environment

Results in the Quarterly Progress Report for July-August-September 1967 showed little agreement between cold-trap temperature,

plugging-indicator temperature and chemical analyses for oxygen. Although the system was being cold-trapped at 250 F, the plugging indicator showed at least two distinct breaks and oxygen values ranging from 80 to 438 ppm O₂ were obtained by chemical analysis. Additional cold trapping during this past report period has apparently removed extraneous impurities from the system. The plugging indicator now shows only one break, and chemical analyses show an oxygen content of <10 ppm. Metal tabs are exposed to determine the carburization characteristics of the system, and the following schedule has been initiated:

O ₂	Weekly (single analysis or as required)
C	Semi-monthly (single analysis, filtered)
H ₂	Monthly (single analysis)
Off-gas	Monthly (single analysis)
Emission Spectrography	Semi-monthly (single analysis)

Loop 2 - Oxygen Contaminated Sodium Environment

A comparison of the plugging-indicator calibration curve and the Los Alamos oxygen solubility curve was made in the last quarterly. Since that time, additional data were procured from Los Alamos; the actual data points are shown in Table 3. The plugging indicator calibration curve and the Los Alamos solubility data are plotted in Figure 1. Those data in Table 3 marked with asterisks are not included in the curve. Agreement between the two curves seems to be within experimental error.

Specimens are now being exposed in Loop 2. A sampling schedule identical to that of Loop 1 has been instituted.

Loop 3 - Clean Sodium Environment

Efforts are underway to calibrate the plugging indicator of Loop 3. At a cold-trap temperature of 250 F, the plugging indicator shows saturation temperatures ranging from 220 F to 255 F; chemical analyses indicate 6 and 9 ppm O₂. Additional samples will be taken at different cold-trap (plugging-indicator) temperatures. The sampling schedule for Loop 3 is identical to that for Loops 1 and 2.

Static Retorts

All four retorts have been sampled with the cold trap and all show <10 ppm O₂. An off-gas analysis of the sodium in

TABLE 3 - SOLUBILITY OF OXYGEN IN SODIUM - LOS ALAMOS

	125	150	165	175	185	200	215	225	250	265	275	300
Oxygen Concentration (ppm)	1.7*	2.3	4.0	4.9	6.8	8.1	10.6	21.9	37.1	46.5	74.4	145.9
	1.7*	2.5	4.6	4.9	6.8	9.0	11.6	20.1	40.1	48.7	72.3	134.5
	2.3*	2.3	3.3	5.0	7.2	7.9	11.2	18.5	37.8	49.2	74.3	145.3
	2.5*	2.0	3.7	4.6	7.5	9.5	13.1					
	2.2*			5.4	6.3							
	1.7*				6.7							
	1.7											
	1.4											
	1.4											
	1.2											
Average Concentration (ppm)	1.8	2.3	3.9	5.0	6.9	8.6	11.6	20.0	38.0	48.0	74.0	142.0
Standard Deviation (ppm)	0.4	0.2	0.5	0.3	0.4	0.8	1.0	2.0	2.0	1.0	1.0	6.0
Relative Standard Deviation (%)	22.0	9.0	13.0	6.0	6.0	9.0	9.0	10.0	5.0	2.0	1.0	4.0

* Initial samples taken during preliminary system checkout.

FIG. 1 - COMPARISON OF MSAR PLUGGING INDICATOR CALIBRATION DATA vs LOS ALAMOS SOLUBILITY CURVE

Static Retort No. 4 showed 2 ppm C_2H_2 , 23 ppm CO_2 , and 3.7 ppm at mass 43. The sampling schedule for the retorts is as follows:

O_2	Semi-monthly
C	Monthly (filtered)
H_2	Monthly
Off-gas	Monthly
Emission Spectrography	Monthly

2. EXTENSOMETER DEVELOPMENT PROGRAM - D. A. Green

Two completed LVDT's were cycled, frequently to 1200-1300 F. These LVDT's incorporated all the latest design and material changes and achieved the first operation at these temperature levels without material and/or circuitry failures. This success then permitted attention to turn to other study areas, such as core quality, core position, sensitivities and stability.

During the testing, the two new units had been connected to operate as compensating units but failed to do so. Independent variables during the heating period were present and not yet identified.

Attention was focused on a single unit determining sensitivity in terms of core movement. The commercial demodulator applied 2.6 volts to the primary at 1600 HZ. Maximum amplification was required to achieve a full-scale reading on the Micromax Recorder for 3/8 in. core movement.

A similar test was made using a power amplifier to boost the primary to 10 volts. A corresponding increase in secondary output was still considered unsatisfactory.

Again, a test was made using the commercial demodulator (oscillator portion only) and amplifying the LVDT output using a single stage Class B transistor push-pull network and rectifying the output. This time, good sensitivity (25 MV(DC)/0.040 in. core movement) -- well within the requirements -- was achieved. The transistor amplifier produced a clean recording without "noise".

Subsequent heat runs continued to produce unaccountable variations, always increasing directly with increasing temperature. When cooling, some retracing was evident, but not to 100%. It appeared as though the core was moving minutely during heating and during cooling.

Considerable time was devoted to the design and testing of suitable electronic circuits for use with the high-temperature differential transformer. Several problem areas have been isolated and solutions have been outlined as follows:

1. Transformer construction

- A. Wire and insulation (#28 Constantan)
- B. Coil form and size (as previously described)

Two complete assemblies have been made and cycled several times.

2. Erratic read-out at any degree of sensitivity.
Contributing causes are:

- A. Line voltage variations
- B. Demodulator creep
- C. Change in LVDT impedance

Tests were run to eliminate line voltage variations by building a separate battery-operated oscillator-receiver system. The sensitivity of this system was such as to indicate a core movement of 0.001 in. per 1.0 mv. Heat runs to 1400 F at the null point showed a stability of less than 0.03 mv on a 5.0 mv scale. These tests proved there was no relative core movement during the heating cycle; i.e., neither the core nor the coil moved relative to the housing.

Tests run with the core off null revealed the combined errors of system creep. The electronic error produced a gradual increase in output amounting to 10% over a period of four hours. This error, it is believed, can be corrected. A change in LVDT impedance is mostly an increase in resistance at the higher temperature. This imposes a lesser load on the oscillator-output transformer which then supplies a higher voltage. This increment is amplified and appears in the secondary as an additional increase of 10 to 15%. The correction of this error will involve the addition of a voltage-regulating or compensating circuit good for $\pm 1/2\%$ of the oscillator output voltage.

Further tests at 1200 F for long time periods are necessary to insure stability over time periods that duplicate creep and creep-rupture test periods.

3. DEVELOPMENT OF TECHNIQUES FOR MEASUREMENT OF IMPURITIES IN SODIUM - F. Roehlich

As authorized under Task II, the surface-tension work is being actively pursued. Surface-tension measurements are continuing with the main area of interest being in the pursuit of defining the relationship of the cold-trap temperature (oxygen content of the sodium) and the surface tension.

The surface tension apparatus was drained and cleaned, and the inoperative electrical gland was removed and replaced. The capillary tube tips were cleaned, polished, and squared in a common plane. A drain hole was drilled into the end plate of the cold leg and a 3/8 in. compression fitting was welded to this orifice. The apparatus was then degassed at 800 F until a vacuum of better than 1×10^{-3} mm Hg was maintained, at which time the unit was charged with approximately 3.6 lbs of reactor-grade sodium. Charging was accomplished through the drain port in the bottom of the cold trap instead of through the sampling port in order to minimize splashing and bubble formation. The following program schedule was then established:

1. Obtain surface-tension data at 700 F with the bottom of the cold trap frozen.
2. Obtain surface-tension data at 300 F with the bottom of the cold trap frozen.
3. Obtain surface tension data at 300 F with the cold trap at 300 F.
4. Obtain surface tension data at 700 F with the cold trap at 700 F.

Pulsations in the manometer level occurred with the chamber temperature at 700 F. This precluded any reliable maximum bubble pressure data. After 24 hours at 700 F, the chamber temperature was reduced to 287 F and the trap to 95 F (at the reference point at mid-trap). Insulation was packed around that portion of the apparatus protruding above the furnace in order to further minimize pressure variations at the liquid metal surface. New spacing rods for coupling the micrometer to the immersion tubes were machined. Surface tension values obtained with the trap frozen and with the trap at 300 F were as follows:

<u>Date</u>	<u>Chamber Sodium Temperature</u>	<u>Cold Trap</u>	<u>Surface Tension</u>
12/18/67	287 °F	95 °F	180 dy/cm
12/19/67	290	105	185
12/20/67	290	107	189
12/21/67	332	327	223
12/22/67	338	329	214
12/26/67	325	318	220
12/27/67	327	323	224
12/28/67	290	105	217*
1/ 2/68	292	108	207 178*

The readings through December 27 indicated reproducible changes in surface tension with corresponding cold-trap temperature changes. The data have been evaluated and are judged to represent real changes in surface tension over and above minor changes from any temperature changes of the bulk sodium. The latter readings (*) were not reproducible and the trouble was attributed to the inability to determine a reliable liquid level directly by using the electrical probe. The probe indicated one level, while observation of the manometer while immersing the probe indicated another.

Visual examination of the sodium surface showed the presence of a surface layer of impurity. Several additional measurements were then made using the manometer level as a surface indicator, but the readings were not reproducible. An attempt is presently in progress to remove the surface interference by aging the sodium at a chamber temperature of ~ 700 F with the cold-trap temperature below 200 F.

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