

Liquid Sodium Technology Development. (I)
Test loops, purification methods and
Supplementary techniques

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Liquid Sodium Technology Development (I)

Test loops, purification methods and supplementary techniques

Summary

The initial results of the development program of the liquid sodium loops and their associated technology are reported covering until the end of 1965. First the three test loops, i.e. corrosion test loop, purification test loop and Mother and Daughter test loop system, and their operating experiences are described.

In Mother and Daughter test loop system, various tests are made with Daughter test loops, using sodium with given contents of impurities which are controlled in the Mother loop. Then, the results of development and its operating procedure are described for the purification system (cold trap, cold finger and hot trap). The first model of the cold trap has a sodium-oxide removal-capacity of 1.3 kg/30 l. Improvements to this trap are proposed for increasing the capacity to three time the initial one by controlling its internal temperature distribution. The results of corrosion and carburization tests are also shown. In the tests, emphasis was placed on austenitic stainless steels (304, 316, 347 S.S.), followed by Cr-Mo steels and refractory metals (Nb, Ta, Mo, Zr and Ti). The analytical data and behaviors are also described for oxygen, carbon, etc., in sodium.

The results of developments, and the cautions to be taken in their operation are shown for various instruments (impurity-, level-, pressure- and flow-meters), preheating system, component recleaning method, and hazard protections (sodium handling, protective clothes and fire extinguishing).

Nov., 1965

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液体 Na 技術開発 (I)

試験ループ、精製法および補助技術

要 旨

液体 Na loop 技術および関連技術の初期開発成果のうち 1965 年末までの成果を報告した。

まず、使用された 3 本の試験ループ——すなわち、腐食試験ループ、精製法試験ループ、および mother and daughter 試験ループ系——の概要と運転経験を述べた。Mother and daughter 試験ループは、mother loop で純度管理された一定不純物濃度の Na を用いて、数本の daughter test loop で各種の実験をおこなう構成のものであり、技術開発にきわめて有用であった。

次にループ内 Na 用精製装置 (cold trap, cold finger, hot trap) の開発結果および標準運転法を示した。1st model の cold trap 酸化物捕獲性能は酸化物として 1.3 kg/30 l であり、これはさらに約 3 倍改善されることが結論された。その改善策の一つは、温度分布の可変制御である。

腐食試験および浸炭試験結果も報告した。試験は主として austenitic stainless steel (304, 316, 347 S.S.) に向けられたが、あわせて Cr-Mo 鋼および耐熱合金 (Nb, Ta, Mo, Zr, Ti) も取上げた。

また Na 中の酸素・炭素等の分析法、反応挙動についても報告した。

種々の装置 (不純物計、液面計、圧力計、流量計) 予熱法、機器洗滌法、防災法 (Na 取扱法、防護具、消火法等) に関しても、開発成果を示した。

1965 年 11 月

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1. Introduction

The development of sodium technology in Japan was started by Hitachi Ltd. in 1957 and the joint research of Hitachi and the Japan Atomic Energy Research Institute (JAERI) was started from 1960. These efforts have resulted in mastering of some of the basic techniques¹⁾. With an increased recognition in Japan of the importance of the fast breeder reactor development, new research facilities were established in JAERI in the summer of 1965 and a full scale development program of the basic technologies was originated. As an intermediate goal, 1~2 MW heat transfer component test loop to test the overall system technology is being planned. With this development program the technology will be available to construct various large sodium test facilities required when, in the future, the experimental reactor plans become a reality.

In the following sections, the results of the development effort led by JAERI will be reported. Initially the general description of the three test loops used in the past and being used today will be given. In Sec. 3, the basis of the sodium loop technology, the purification method of the loop sodium, will be described. In Sec. 4, the results of the preliminary corrosion test will be reported briefly. In the next section, Sec. 5, the other supplementary technologies and component developments, i.e. chemical analysis of sodium metal, impurity meter, level meter, pressure-gage, flow-meter, system preheating and recleaning, hazard protection, etc., will be outlined. Detailed reports will be given in the future articles.

2. Sodium test loop

2.1 Corrosion test loop

This loop was designed by the cooperation of Hitachi and JAERI and constructed in the Hitachi Research Laboratory in January, 1961, according to the joint research contract signed in January, 1960 between JEARI and Hitachi Ltd. The loop was used in research up to November of 1963. At the time, a plan for sodium cooled reactor development project was not contemplated and therefore the research objectives were high temperature, high velocity mass transfer test and semi-static corrosion test. But in practice, the loop was useful as a training facility for the preliminary Na and NaK handling and loop operation techniques.

The loop piping was type 304 stainless steel tube (25.4 mm O.D. \times 2 mm thick) and the loop consisted of a electromagnetic pump (A. C. Faraday type, 20 g.p.m. maximum), a permanent magnet

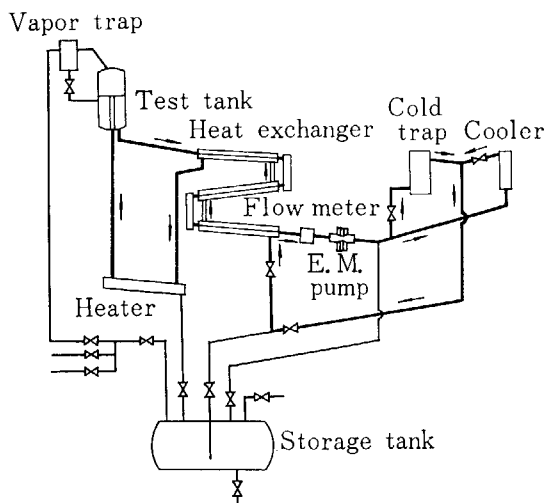


Fig. 1 Flow sheet of the corrosion test loop

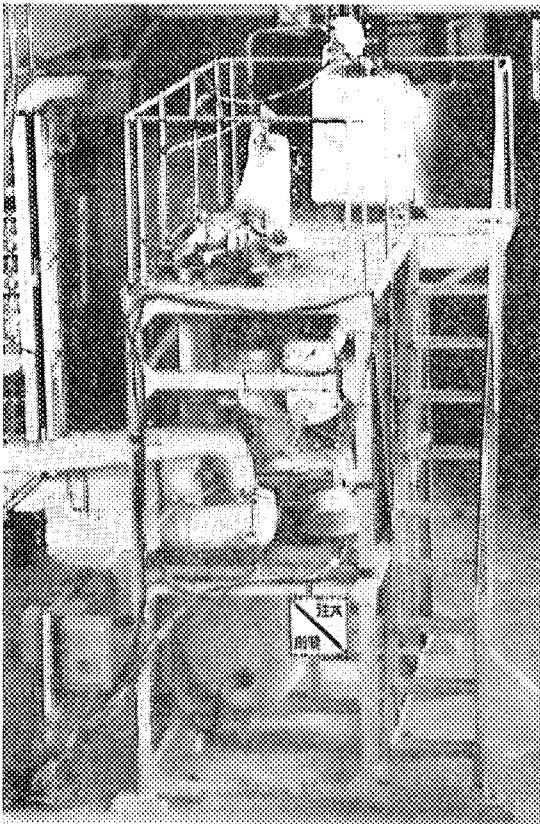


Fig. 2 General view of the corrosion test loop

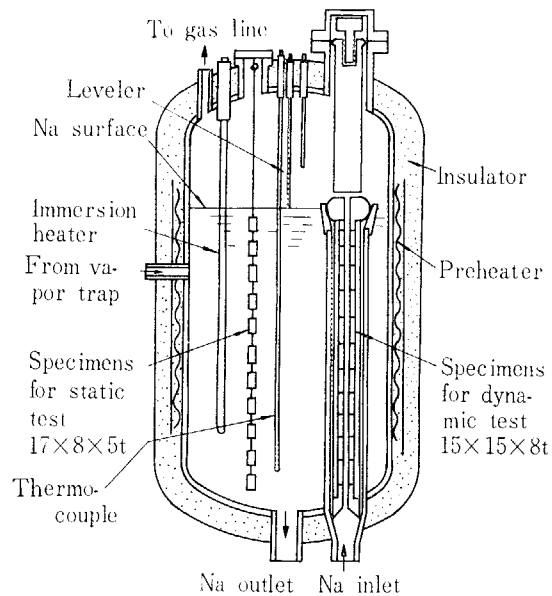


Fig. 3 Test section in the corrosion test loop

flowmeter, main heaters(20+15kW), an economizer, a cooler, a cold trap, an expansion tank, a storage tank, and valves. (c.f. Fig. 1 and Fig. 2). Inside the expansion tank, there is a high flow rate test section with a maximum flow rate of 10 m/sec (cross section 2 mm \times 10 mm) and a semi-static test section. (c. f. Fig. 3). The maximum test temperature was 600°C. As can be seen from TABLE 1, six runs (1490 hours in total) with NaK (Na-44 w/o K) and four runs (1653 hours in total) with Na were carried out. As stated previously, a preliminary corrosion test was performed and a part of the results obtained with the JAERI specimens (mainly 300 series austenitic stainless steels) will be reported in Sec. 4.

The conclusion reached as a result of these experiments was that NaK was more difficult to handle than Na.

2.2 Purification test loop

With the lead of JAERI, the corrosion loop described in the previous section was almost completely modified and converted into a developmental test loop for a cold trap, a hot trap and a plugging indicator. After the completion of the facilities in the August of 1964, the experiments were carried out with the cooperation of Hitachi up to June, 1965.

The loop flow sheet and the general view are shown in Fig. 4 and 5. A cold trap, a hot trap, a hot trap economizer, a plugging indicator, an expansion tank (with two viewing windows, an induction-coil type level meter and a vapor trap), a freeze trap, bellows valves and freeze-seal valves, etc. were either added or replaced with a new one. The total length of the piping was approximately 50 m and the total volume was about 150 l. With the exceptions of the hot trap and the hot trap economizer which were made of type 310 stainless steel, all other parts were made of type 304 stainless steel.

As can be seen from TABLE 2, three runs(1132 hours in total) were performed and besides the

TABLE 1 High temperature, high velocity corrosion tests

Run	Date	Period (hr)	Specimen (Prepared by JAERI)		Temperature (°C)	Velocity of Na (m/sec)	Cold trap Temp. (°C)	Note
			Dynamic	Static				
NaK-1	Apr. 1961	150	303 S. S. Cr-Mo steels (4 kinds)	304 S. S.	600	8	180	
NaK-2	Aug. 1961	170	304 S. S. Cr-Mo steels (2 kinds)		600	4	160	Heater power up; Removed of damaged valves; Stabilizer added
NaK-3	Sep. 1961	215	304 S. S. Cr-Mo steels (4 kinds)		600	4	160	Na leakage from E. M. pump
NaK-4	Jan. 1962	170	304 S. S. Cr-Mo steels (1 kind)		600	4	170	
NaK-5	Mar. 1962	720	304 S. S. Cr-Mo steels (2 kinds)		600	4	160	
NaK-6	Jun. 1962	63	304 S. S. Cr-Mo steels (2 kinds)		600	8	180	Na leakage by preheater short
Na-1	Oct. 1962	133	304 S. S. 316 S. S. 347 S. S. Cr-Mo steels (3 kinds)		600	10	200	Na leakage from main heater No. 1; Exchange of cold trap and specimen holders; Main heater (No. 2) added
Na-2	Feb. 1963	400			600	10	210	Repair of main heater No. 1; Na-overflow into gas line
Na-3	Jun. 1963	720			600	10	210	
Na-4	Nov. 1963	400	Nb, Ta added to abovementioned specimens		600	10	200	Dismantled of cold trap, cooler and test tank
		Total 3141 hr						

TABLE 2 Purification tests

Run	Date	Period (hr)	Object of test	Note
1	Sep. 1964	394	1. Performance test of cold trap 2. Performance test of plug. ind., lower and observing window	Reconstruction of plugging ind. Setting of hot trap
2	Nov. 1964	0		Failure of Na filling
3	Feb. 1965	166	1. Performance test of cold trap 2. Performance test of plug. ind.	Na leakage from heat economizer and its repair.
4	May 1965	572	1. Performance test of hot trap 2. High temp. corrosion test	Disassembly and analysis of cold and hot traps
		Total 1132 hr		

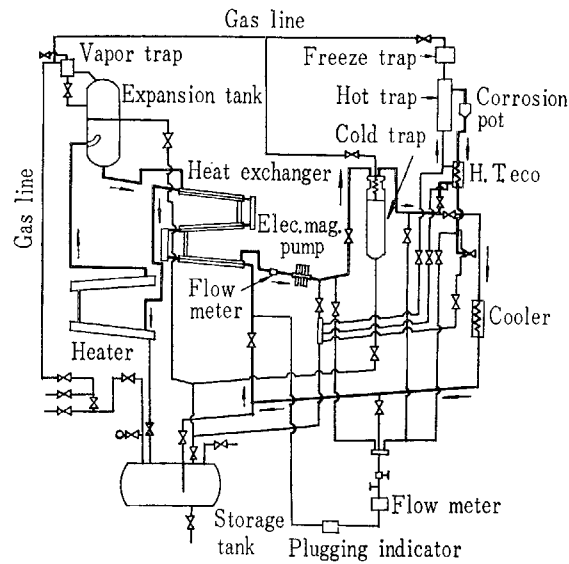


Fig. 4 Flow sheet of the purification test loop

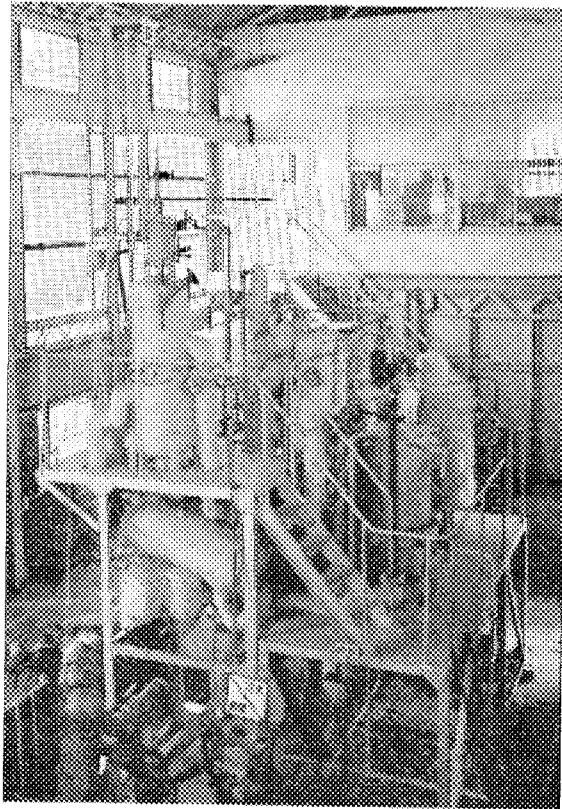


Fig. 5 General view the purification test loop

improvement in the purification technique, improvements in various supplementary techniques were carried out as shown in this paper.

2.3 Mother (high purity sodium supply) and Daughter test loop system

As the research was being carried on using the purification test loop, the first loop to be set in the new sodium laboratory building at JAERI was designed in the summer of 1963. Because of the delay in the completion of the laboratory building, the loop was not completed until November, 1965. The test operation was started immediately and at first, various techniques developed in the past were improved and integrated. Then a series of research and development projects were started and are now being carried out.

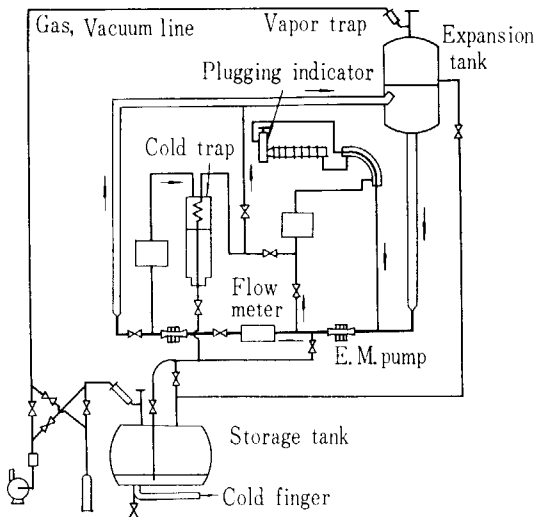


Fig. 6 Flow sheet of the Mother loop

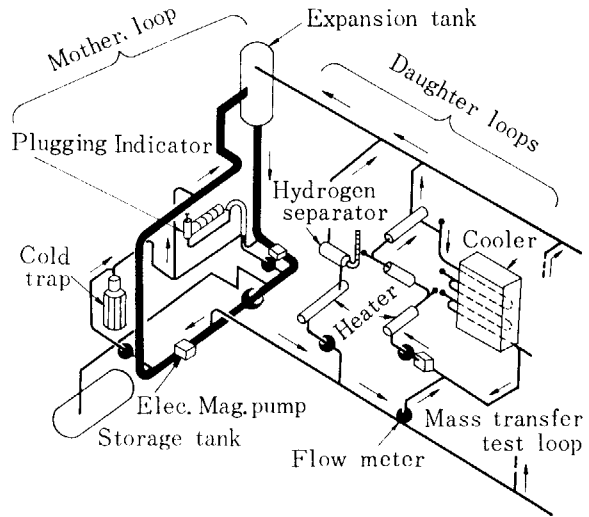


Fig. 7 Schematic of the Mother and Daughter test loop system

Although the main purpose of the Mother loop was to supply the high purity sodium to the Daughter test loops, the Mother loop will probably serve as the base for the 3" pipe loop technology, which corresponds to 3 MW loop technology in a thermal scale, and other developments.

The Mother loop is shown in Fig. 6 and 8., which occupies the space of approximately 3 m \times 7 m and 8 m in height. The loop has 14.5 m of the 3" main piping (89 mm O. D. \times 4 mm thick) and 38 m of the 1" piping. These piping have an inclination of more than one degree for the perfect dump of loop sodium. A 500 l capacity storage tank has been equipped with an induction-coil type level meter, a vapor trap and a cold finger (c.f. Sec. 3.2). The expansion tank attached to the loop has an effective maximum capacity of 350 l and is equipped with a level meter, two windows, a vapor trap and a sampler. The cold trap in the bypass of the Mother loop is designed for the maximum flow rate of 10 l/min and has the oxygen removal capability of 1 kg (c.f. Sec. 3.1). The loop is also equipped with a plugging indicator with a heat economizer, two electromagnetic pumps a G.E. product of capacity 20 gpm at 20 psig and a MSAR product of 30 gpm at 20 psig, three flowmeters, 16 bellows seal valves, and flexible sheathed electric heaters of about 150 kW in total power for the system preheating. To achieve several flow patterns and to avoid freezing of a stagnant part, the preheating system is of a quite flexible design (c.f. Sec. 5.6). The total number of chromel-alumel thermocouples attached is about 170 including the spares. The temperatures of 36 of them are recorded on chart. The system is composed completely of type 304 stainless steel.

At present, with the minimum expansion tank volume of 150 l, it is possible to provide 200 l of sodium at the plugging temperature of 110°C to the Daughter test loops. In the future, another storage tank of the same capacity as the present one will be added, enabling the Mother loop to provide total of 700 l.

As the Daughter loops, a mass transfer test loop and a hydrogen detection and separation loop were constructed. To these will be added a heat transfer test and a valve test and a flowmeter calibration loop. A schematic figure and a photograph of the Mother loop and the Daughter loop (mass transfer test loop) are shown in Fig. 7 and 8.

The advantages of Mother and Daughter loop system are:

- (1) The system becomes simple because there is no need to have, for each loop, an independent purification system and impurity detection system.
- (2) The same sodium under a well controlled impurity-concentration condition can be used for various experiments parallelly.

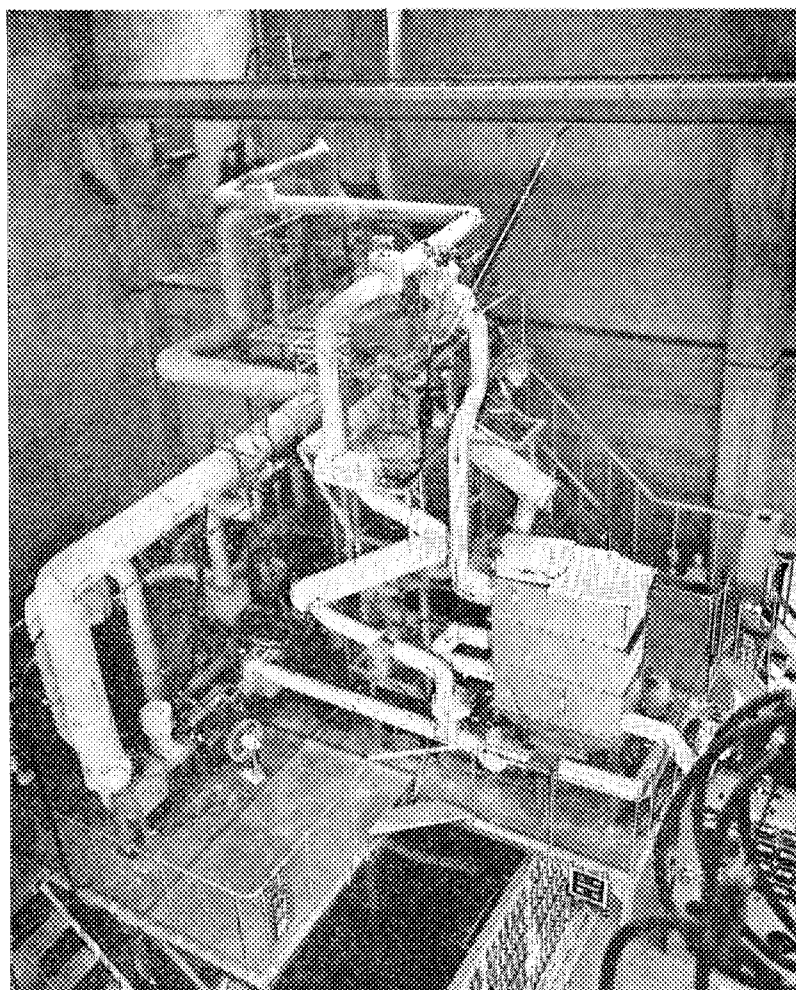


Fig. 8 General view of the Mother and Daughter test loop system (Feb., 1966)

- (3) It is technically easier to operate a series of interconnected loops constantly than to operate many independent loops individually.
- (4) It is possible, after clean up, to isolate a specific Daughter loop for carrying out an experiment.

3. Purification methods of sodium in loops

Because excessive impurities, besides having detrimental effects on the material compatibility, cause problems by deposition and plugging in the cooler part of the loop, the maintenance of sodium purity is the most important item of sodium loop technology. The non-metallic impurities that we are presently interested in are oxygen, carbon and hydrogen.

3.1 Cold trap

The oxygen and hydrogen saturation solubility curves that are, at present, judged to be most reliable are shown in Fig. 9. The oxygen curve is based on the equation

$$\log_{10} (\text{w/o oxygen}) = 1.21 - \frac{1777}{T(^{\circ}\text{K})} \quad (1)$$

proposed by Claxton²⁾ for the range of 110~555°C. The hydrogen curve is based on

$$\log_{10} (\text{w/o hydrogen}) = 6.211 - \frac{5021}{T(^{\circ}\text{K})} \quad (2)$$

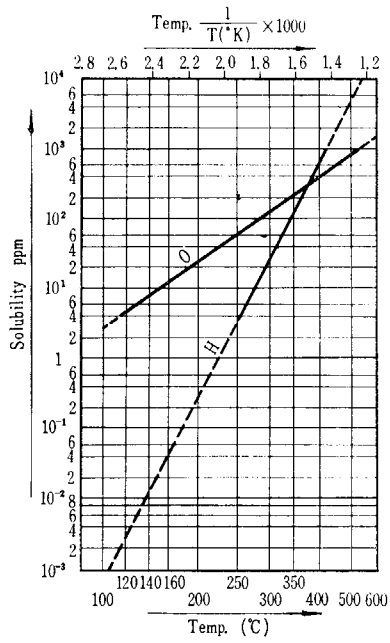


Fig. 9 Solubility curves of oxygen²⁾ and hydrogen³⁾ in sodium

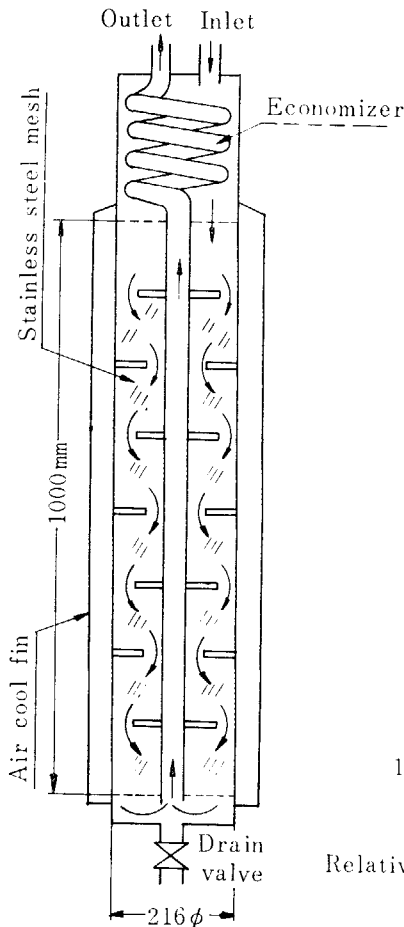


Fig. 10 Cold trap of the Mother loop and trapped oxygen distribution

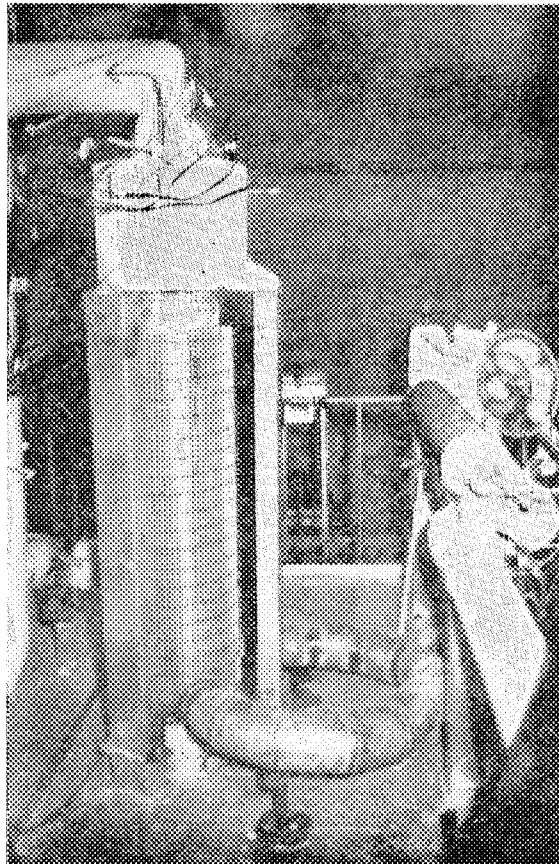
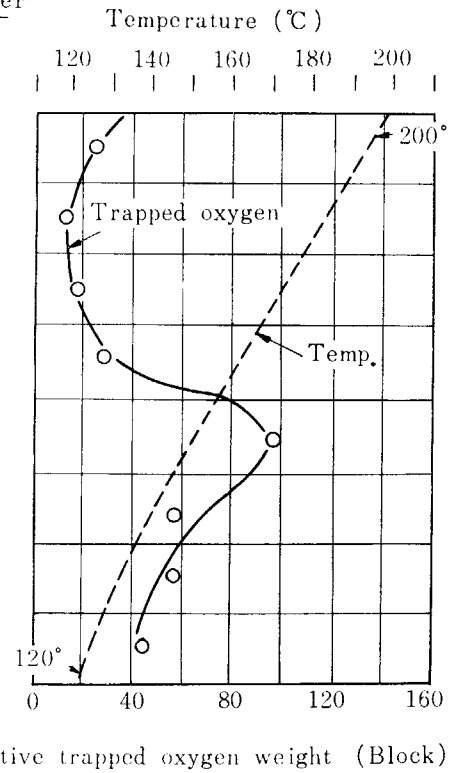


Fig. 11 Cold trap with the cover removed

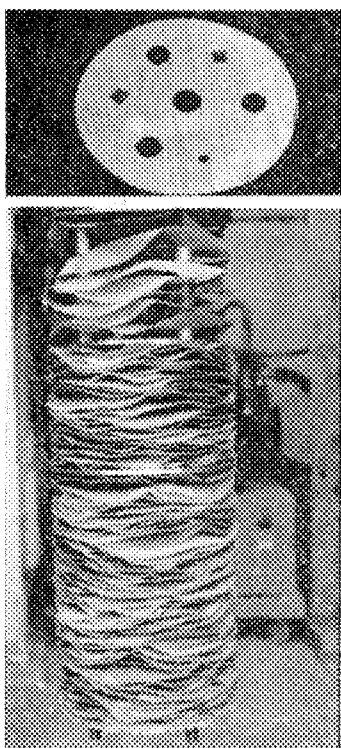


Fig. 12 Packed mesh and its assembly of the cold trap

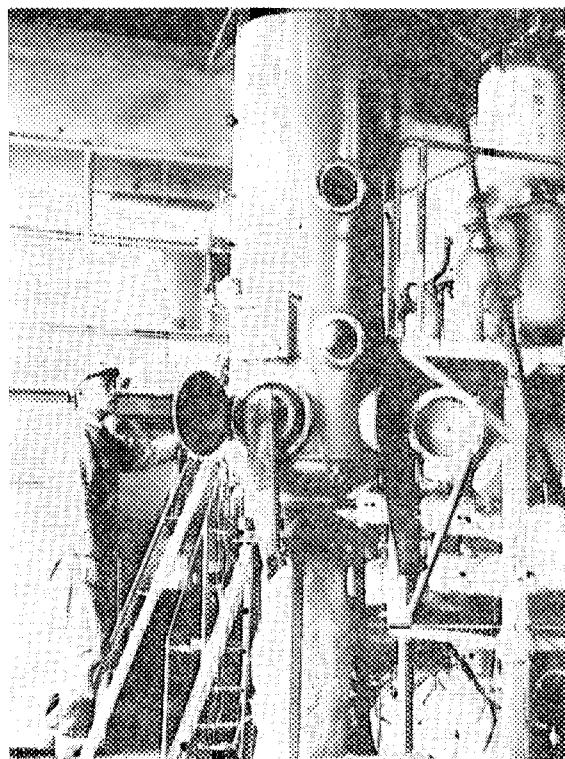


Fig. 13 Special glove box for the disassembly of components

estimated by ADDISON *et al.*³⁾ for the range of 250~450°C. For carbon, there is no reliable data but in sodium with oxygen content of 40 ppm, the solubility is reported as approximately 20 and 70 ppm at 130°C and 500°C respectively.⁴⁾ Thus it is possible to separate these elements by cooling sodium nearly to the freezing point.

The cold trap developed for the purification test loop is shown in Fig. 10 and 11. The trap is a cylinder with an effective capacity of about 30 l (200 mm I. D. × 100 mm high). The main features of the design are:

- (1) To prevent plugging in the dirty inlet pass, a Hallam type helical internal economizer⁵⁾ with wide inlet pass was used.
- (2) As the packing material in the deposition region, waved type 304 stainless steel mesh of 30~40 mesh and 200 mm diameter were coarsely packed at 3~4 mm interval. Furthermore, two or three holes of 20 mm × 20 mm are opened at random in the screen, and the trap was packed alternately with buffer plates and screens to form a complex flow path for sodium. The resulting packing density was approximately 0.18~0.25 gr/cm² (see also Fig. 12).
- (3) The cooling of the outer face of the cylinder was by forced convection air cooled fins and in addition, to permit an easy control of the internal temperature, flexible sheathed heaters were coiled around the cylinder (c. f. Fig. 11). Use of oil burner or liquid coolant is undesirable because it makes the temperature control difficult and thus causes large thermal stresses due to sodium freeze-melt cycle. The previously mentioned helical economizer is effective in reducing this stress.
- (4) The upper part is of simple design with welded flange type without cover gas. This permits cold trap regeneration and visual observation by simply removing the trap from the line, cutting the above mentioned flange, connecting the trap to the lower part of a special glove box and withdrawing the internal structure into an inert gas atmosphere after heating to about 200°C. Then, after cooling, various experimental operations can be carried out in safety. (c. f. Fig. 13).

From various experiments, the following became clear :

- (1) When the trap was operated at a flow rate of about 4 l/min with Na_2O_2 supplied continuously from the expansion tank to keep the oxygen concentration at 10~50 ppm, the total amount of impurities removed by the trap up to the time of plugging of the trap was equivalent of 1.3 kg of Na_2O (equivalent to 340 gr of oxygen). This value was estimated from the weight of the trap after disassembly, dumping, and cooling inside the glove box, and from the weight change and chemical analysis of the eight standard mesh specimens that were inside the trap. The average temperature distribution and the difference in the oxygen removal capabilities of various parts of the trap are shown in Fig. 10.
- (2) For the flow rate of less than 10 l/min., the effectiveness of the impurity removal by a single pass was 100%. That is, the minimum temperature of the cold trap agreed with the plugging temperature of sodium.
- (3) From these experiments, it was concluded that our design concept was basically sound and required no modification. But to permit more flexible operation and to increase the removal capacity to three times the present capacity, the following improvements are recommended :
 - (a) Add an external economizer.
 - (b) Control the internal temperature distribution to agree at all times to the current plugging temperature and to reduce the temperature gradient to a minimum. (c. f. Fig. 17)
- (4) As discussed in Sec., 3.5, this trap can be used in a loop with a maximum sodium volume of 3000 l. There is a possibility for scaling up the trap capacity further.

3.2 Cold finger of storage tank

Since sodium inside a storage tank normally would not be purified, the sodium inside the tank may gradually become contaminated. For this reason and for extending the life of the cold trap, a natural circulation cooled, diffusion cold trap (cold finger) was attached to the bottom of the storage tank. As can be seen from Fig. 14, it is a 3" (89 mm O.D. \times 4 mm thick) bare pipe

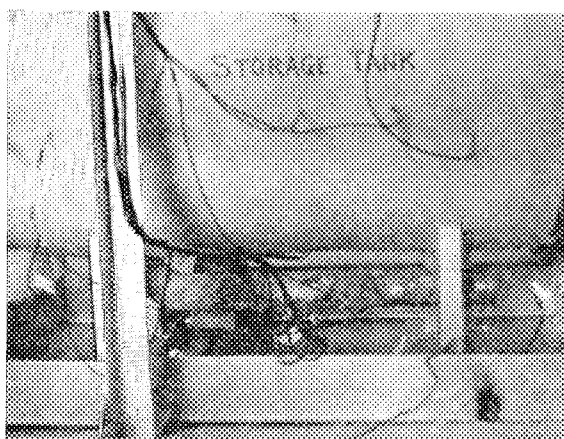


Fig. 14 Cold finger of the storage tank

of about 80 cm length welded to the tank. With the storage tank at 250°C, the temperature at the extremity of the cold finger is 120°C. As the plugging proceeds from the end of the cold finger, the temperature distribution of the cold finger changes and from this change the life of the cold finger can easily be known. At the end of the cold finger life, as much of sodium as possible will be transferred to the loop and as the loop is operating, the remaining sodium will be dumped from the drain valve at the bottom of the storage tank. Then the old cold finger will be cut off and a new one will be welded into place.

The effect of a cold finger can be listed as follows:

- (1) Clean sodium can be supplied to the loop and this will cut the time required for in-loop sodium purification.
- (2) Since, at the time of dumping sodium from the loop, cold trap temperature can be raised to about 300°C, more complete dumping occurs and furthermore, oxide in the trap can be partially removed, e. g. about 10 gr. of Na₂O can be removed if 100 l of sodium passes through the trap.
- (3) As a result of (1) and (2), the life of the cold trap can be extended, decreasing the frequency of the cold trap regeneration operation.
- (4) Since the cold finger is at the lowest point, it is useful in removing the heavy suspensions.

3.3 Hot trap

An experiment to determine the engineering design standard of a zirconium-foil filled hot trap and to determine the effectiveness of using Ti-Zr alloy foil which is cheaper than Zr foil was conducted using the purification test loop. Also, a preliminary batch test of the carbon removal ability of zirconium was carried out using a test pot. (c. f. Sec. 4.3).

The hot trap is a type 310 stainless steel cylinder (140 mm O.D. × 1050 mm high) of 3.85 l (100 mm I.D. × 720 mm high) capacity. Corrugated zirconium foil of 0.1 mm thick and 100 mm wide with waves of 2.5 mm high was wound into a roll with alternating layers of 0.05 mm thick plane zirconium foil. Seven of these rolls were used in the trap (see Fig. 15 and 16). Total of 3.23 kg of zirconium was packed at the density of 0.84 g/cm³. The total surface area was 13 m². The hot trap was equipped with an external, electric heater of 6 kW and at the exit end of the trap, 15 mm × 20 mm × 1 mm specimens of pure zirconium, 80 (w/o) Zr-Ti, 60 Zr-Ti, 50 Zr-Ti, 25 Zr-Ti and pure Ti were placed. The hot trap economizer which also served as



Fig. 15 Hot trap and its economizer (left)

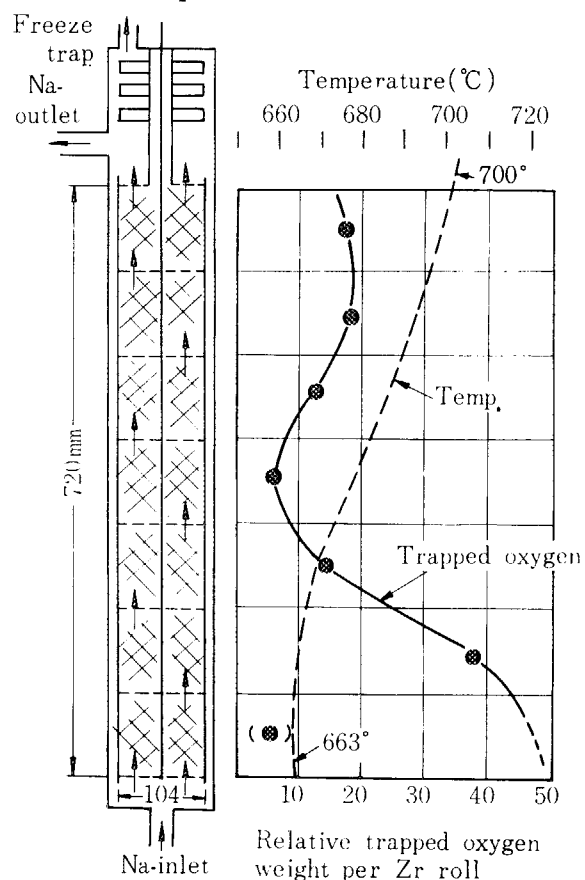


Fig. 16 Schematic figure of the hot trap and getterd oxygen distribution

the heater was a tube-in-tube type (an outer tube 42.7 mm O.D. \times 2 mm thick and an inner tube, 25.4 mm O.D. \times 2 mm thick), made of type 310 stainless steel, with the total length of 15 m coiled into a helical shape. The economizer was heated by a propane burner to bring sodium inlet temperature to 650°C.

The test of the hot trap was carried out at 650~700°C with a series connected cold trap at the minimum temperature of 160~180°C (4~6 ppm oxygen from Fig. 9). At the exit from the hot trap the plugging temperature of sodium was 130~150°C (2~3 ppm oxygen). As with the cold trap, the hot trap was dismantled and withdrawn, for observation, into the glove box after 300 hrs of continuous operation. The results of the hot trap experiment were:

- (1) The reaction product on the surface of zirconium was monoclinic ZrO_2 , but was light black color and it may have been a hypostoichiometric compound.
- (2) The total amount of trapped oxygen was about 60 g and the average oxygen removal rate was about 0.2 g oxygen/hr (15 mg/hr. m^2 of zirconium surface).

In Fig. 16, the relative trapped oxygen weight per Zr roll is shown. In this figure the reason for the abnormally low trapped oxygen weight at the lowest Zr foil is as follows. This foil was near the entrance and was brittle due to heavy oxidation and a microscopic examination revealed cracks and separations of the oxide films. Thus at the time of cleaning, there is a possibility that part of the oxide fell off.

- (3) Zirconium surface was not very effectively used. There was noticeably more oxidation near the entrance and along the central axis of the trap. (This effectively used part was approximately 20% of the total). The contact area between the flat zirconium foil and the waved foil was hardly oxidized at all. As a result, about 40% of the total surface area was not effectively used. There is, therefore, a need to devise baffles or some other means to make the sodium flow more uniform and also a need to minimize the contact area between the foils.
- (4) From the results of microscopic observations and micro-Vickers hardness tests on the zirconium test specimens, the oxide film was found to be stable up to a thickness of about 5 μ , above which the film became brittle and separated from the foil.
- (5) In this series of experiments, oxide films did not adhere to the surfaces of Zr-Ti alloys. The oxidation rate increases with increasing Ti content, but oxide films' ability to adhere to the surface diminishes. If a small quantity of titanium is added, no saving in cost would result, and if a large quantity of titanium is added, oxide films separate and contaminate sodium. Also large quantities of titanium would make rolling of a foil more difficult. Therefore from the overall view point, the use of pure zirconium was judged most practical.

3.4 Procedure for cleaning and purity control in loops

Trouble-free operation of a sodium loop can be said to depend mainly on sodium purity control. What follows is the standard procedure developed from our experiences. Problems are: (a) Acid cleaning and washing before the pipe welding. (b) Cleaning after assembling. (c) Degassing by preliminary heating before sodium charging. (d) Cleaning of sodium in the storage tank by the cold finger. (e) Initial cleaning in the loop. (f) Purity monitoring. (g) Operation at the time of dumping. (h) Regeneration of the purification apparatus. In this paper, (c), (d), (e), (f), (g) and (h) will be discussed. The operation of the hot trap will not be discussed.

- (1) At first, sodium in the storage tank is heated to 250~300°C. By maintaining this condition for at least a half day, the cold finger of the storage tank will trap some impurities and solid suspensions will be allowed to settle out. The piping is degassed to 10^{-3} mmHg as it

undergoes the preliminary heating.

- (2) The sodium is charged slowly into the loop to avoid sodium velocity in excess of 2~3 m/sec in the piping. As soon as the level of sodium in the expansion tank becomes the predetermined value, the drain valve is closed, the pump started and preheating terminated. Next, the plugging temperature is determined by the plugging indicator and the temperature distribution appropriate to that plugging temperature is established inside the cold trap. When the plugging temperature becomes less than 150°C, the loop is cleaned by raising the loop temperature gradually, taking care not to allow the plugging temperature to exceed 170°C.

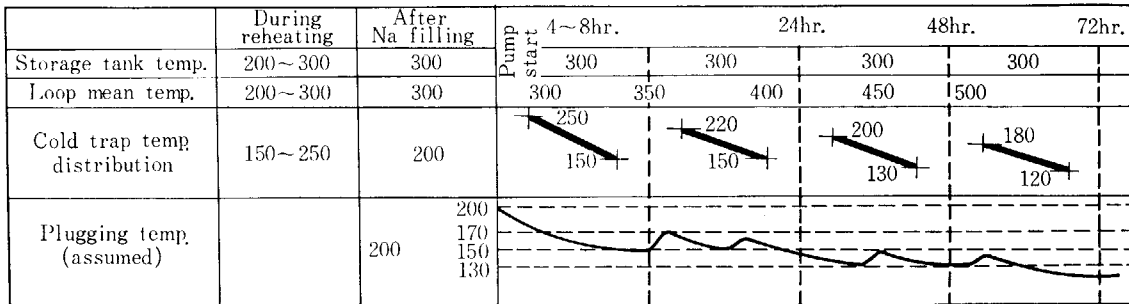


Fig. 17 Cleaning procedure for sodium in loop

This model procedure is shown in Fig. 17. In the case of the first cleaning of the Mother loop with the fresh 304 stainless steel surfaces, it required about three days near 400°C and one day at 500~550°C in loop temperature before the plugging temperature stabilized at 110°C. It is desirable to maintain a flat temperature distribution in the cold trap after the loop operation reaches steady state. With our present cold trap, this adjustment is quite simple. But considering the possibility that, due to an accidental contamination, the plugging temperature of the loop might suddenly rise, it is necessary that the cold trap temperature be set so that the highest temperature in the cold trap does not become lower than the plugging temperature of the loop sodium. Except for a very small system, the highest temperature in the cold trap can be 180°C and the lowest 110°C.

- (3) The impurity removal time for the whole system, from the practical consideration, should be less than 5~6 hours. The factors determining the required removal time are the total quantity of sodium in the loop, $W(l)$, and the volumetric flow rate, $w(l/min)$. If the initial concentration in the loop, the final concentration in the loop (corresponds to the lowest temperature in the cold trap), and the concentration at time t , are taken as x_o , x_f , x in weight percents respectively, it is well known that the following equation holds⁶⁾.

$$X = \frac{x - x_f}{x_o - x_f} = e^{-\frac{w}{W}t} \quad (3)$$

and

$$X = \frac{1}{e} \quad (4)$$

at

$$t_e = \frac{W}{w} \text{ (min)} \quad (5)$$

Thus t_e must be chosen in the range of 50~100 min. For the cold trap described in Sec. 2.3, $W=500 l$, $w=4\sim 10 l/min$, so that $t_e=125\sim 50$ min. The result of the experiment agreed well with this calculation. If we choose $t_e=300$ min and $w=10 l/min$, $W=3000 l$.

- (4) For the purity monitoring, Rhometer is suitable but there also is the possibility of improving the plugging indicator to allow automatic, continuous monitoring at a plugging temperature as low as 105°C.

- (5) At the time of dumping, the drain valve at the bottom of the cold trap is also opened and the whole cold trap is allowed heat up to 300°C. By this mode of operation, if 100 kg of sodium containing 100 ppm of oxygen is dumped, 10 gr of oxygen will be removed from the cold trap. This oxygen will probably transfer to the cold finger of the storage tank.
- (6) If and when the cold trap is plugged, it must be regenerated. For this, sodium must be melted for disassembly but if the inner assembly is taken out at high temperature, besides risking sodium combustion there is a danger of damaging the materials. Therefore, first, the trap is removed from the piping system and, as described in Sec. 3.1, the inner part is withdrawn into the inert atmosphere of the glove box. Then, after cooling to room temperature, the trap should be taken out into the ordinary atmosphere for steam recleaning. Problems remaining to be further studied include effective removal and cleaning methods for carbon, carbon compound, and metallic suspensions. For up to 5 μ , a stainless steel filter is effective but for less than 5 μ , vacuum distillation method may become practical.

4. Preliminary corrosion tests and impurity behaviors

The tests described in the following sections, 1, 2 and 3 are the preliminary sodium corrosion tests. Therefore, these tests have only a qualitative meaning but since they are valuable for understanding the behavior of impurities, the general results will be described. To obtain more quantitative data, the mass transfer test Daughter loop, as described in Fig. 7 and 8, was built and the tests are being carried out.

The conclusion reached as a result of these tests is that the austenitic stainless steels and Cr-Mo steels are quite acceptable for the maximum sodium temperature of 600°C, the maximum sodium flow rate of 10 m/sec, and the maximum temperature drop of about 300°C, provided that the impurities are sufficiently removed by a cold trap.

4.1 High velocity mass transfer test

Since 1961, using the corrosion test loop (Sec. 2.1) which is capable of reaching 600°C and producing 10 m/sec (18 l/min) flow rate, the corrosion, erosion, and mass transfer phenomena of various materials in NaK and Na were investigated. The specimens tested and analyzed by JAERI and the conditions of the tests are listed in TABLE 1. The results of the measured weight change, hardness change roughness change, and microscopic observation are considered in detail.

(1) 300 series stainless steels

- (a) The weight changes of various steel specimens are shown in TABLE 3. The difference between the effect of Na and the effect of NaK was not apparent. There also was

TABLE 3 Weight change (mg/cm² month) in the high velocity mass transfer tests

Material		Material			
		304	316	347	2 ¹ / ₄ Cr-1 Mo
NaK	0 m/sec	+0.23~-0.07			+0.33~-0.07
	4	-0.57~-6.7			-0.29~-2.2
	8	-0.05~-12			-5.2~-8.6
Na	0	-0.04~-0.22	0~-0.17	+0.40~-0.37	+0.21~-0.22
	10	-1.1~-7.1	-1.1~-9.0	+0.84~-3.1	-1.2~-24

no meaningful difference between various types of steel.

- (b) The effect of the flow rate was very pronounced. For the flow rates of 0, 4, 8, and 10 m/sec, the corrosion rates were about $\pm 0.1, 1, 7,$ and $10 \text{ mg/cm}^2 \text{ month} = 0.12 \text{ mm/year}$ respectively.
 - (c) The surface roughness was about 1μ and the type 316 stainless steel showed somewhat better result than the others.
 - (d) From the results of the microscopic observation, no preferred corrosion spot, decarburization, or carburization were discovered. Corrosion can probably be considered as proceeding uniformly.
 - (e) No change in hardness could be observed.
- (2) Cr-Mo steels
- (a) 5 Cr-1 Mo steel showed relatively little corrosion weight loss. $2\frac{1}{4}$ Cr-1 Mo steel generally suffered less corrosion weight loss when alloyed with such additives as V, Nb and Ti.
 - (b) From the microscopic observations, no carburization or decarburization was observed indicating apparently uniform corrosion.
- (3) Nb, Ta
- (a) For 0 and 10 m/sec, weight losses of about 20 and 2000 $\text{mg/cm}^2 \text{ month}$ respectively were observed.
 - (b) The surface appeared black and the surface roughness was pronounced. The separation and falling off of the oxide films were notable.

The results, for the different materials described above, were obtained with the cold trap temperature of $160^\circ\sim 170^\circ\text{C}$ for NaK and $200\sim 210^\circ\text{C}$ for Na. The final, steady state value for the oxygen concentration was estimated to be $25\sim 30 \text{ ppm}$ (oxygen). But even under the same test conditions, there were cases where the corrosion losses were different by a factor of two to three. These results may be due to an oxygen contamination during the component repair or due to an insufficient initial loop-cleaning. The effect was greater for faster flow rate and therefore had an important influence in erosion.

After 4300 hours of operation, the corrosion test loop was disassembled and samples taken from the piping. According to microscopic observations, although the surface roughness in the high temperature piping was large (about $3\sim 4 \mu$), the micro-structures were stable and no carburization or decarburization were observed.

4.2 High temperature corrosion test

Test sections were attached to the downstream of hot trap in the purification loop (c. f. Sec. 2.2). The corrosion test of various materials at high temperature (700°C) were carried out in these sections.

The test conditions were; sodium temperature of 700°C , sodium flow rate of $1.3 \times 10^{-3} \text{ m/sec}$, plugging temperature of $140 \pm 10^\circ\text{C}$, and the test time of 300 hours. The specimens were AISI type 304, 316, 347 stainless steels, $2\frac{1}{4}$ Cr-1 Mo steel, 5 Cr-1 Mo steel, pure Nb, Ti, Zr, and Mo. The results were as follows,

- (1) 300 series stainless steels:
 - (a) The weight changes are shown in TABLE 4. The weight loss of 347 was fairly large.
 - (b) From the microscopic observation, no carburization or decarburization were observed. Carbide precipitations at the grain boundaries were observed.
 - (c) By a X-ray diffraction, γ phase was detected in the corroded surface of type 316

TABLE 4 Weight change (mg/cm² 300 hr) in the high temperature corrosion test

Material	304	316	347	
Weight change	-0.055~-0.095	-0.093~-0.097	-0.124~-0.143	
Material	2 ¹ / ₄ Cr-1 Mo (0.6 Si)	2 ¹ / ₄ Cr-1 Mo (0.3 Si)	5 Cr-1 Mo	
Weight change	-0.10~-0.32	-0.36~-0.70	-0.065~-0.32	
Material	Nb	Ti	Zr	Mo
Weight change	-4.0~-5.0	-2.6~-2.7	+1.9	+0.04~-0.004

stainless steel.

(2) Cr-Mo steels

- (a) The weight changes are shown in TABLE 4. No meaningful difference was observed among the different steel types.
- (b) From the microscopic observation, no decarburization was observed.

(3) Refractory metals

- (a) The weight changes are shown in TABLE 4. Nb, Ti and Zr were black from oxidation. In the case of Nb and Ti, the oxide films were found to be separated but no separation of the oxide film was observed for Zr. The surface condition of Mo was not changed from before the test and showed good corrosion resistance.
- (b) Changes were observed in the hardness of Zr and Ti. At 20 μ from the surface, before the test, the micro-Vickers hardness was 200 but after the test it increased to 700. This was probably due to the dissolution of oxygen.

4.3 High temperature carburization test

Tests were carried out to check on the carburization of the materials and the behavior of carbon in sodium. For the test, a type 304 stainless steel pot of about 1 l was used. With the temperature kept at 750°C, Zr foil was inserted and allowed to remain for about 100 hours to remove oxygen and part of carbon. Then the specimens were inserted for the test time of 100 hours. The specimens chosen were AISI 304, 316, 347 and pure Nb. The carbon content of sodium was found to have decreased from about 90 ppm to about 50 ppm.

(1) 300 series stainless steels

- (a) The weight changes at the end of the tests are shown in TABLE 5. In all cases, the result is an weight gain. No meaningful difference among various types of steel was observed.

TABLE 5 Weight change (mg/cm² 100 hr) in the high temperature carburization test

Material	304	316	347	Nb
Weight change	+0.022~+0.03	+0.02~+0.03	+0.022~+0.024	+0.007~+0.027

- (b) By the microscopic examinations about 2~3 μ of carburized layer was observed in every specimen. Carburization was found to be promoted by the grain boundaries and the lattice defects.

(2) Niobium

- (a) At the end of the test, 0.007~0.027 mg/cm² of weight gains were observed.
- (b) By the microscopic observations, no change in the microstructure was observed. There were black carbide film on the surface.
- (c) From the chemical analysis, the carbon concentration was found to have increased from

40~50 ppm to 100~160 ppm.

(3) Zirconium

- (a) The zirconium foil used as the getter for oxygen was found to have 1~2 μ oxide film layer. The total "gettered" oxygen by about 40 g of Zr was about 250 mg.
- (b) From the chemical analysis, the amount of carbon was found to have increased from 120 ppm to 1400~1600 ppm (about 50 mg). It seems that when oxygen has been "gettered", carbon is absorbed.

4.4 Analysis by X-ray micro-analyzer

From among the specimens of the tests of Sec. 4.2 and 3, change in the Fe, Cr, and Ni contents of AISI 304, 316, 347 specimens were checked by X-ray micro-analyzer. A typical example is shown in Fig. 18. Considering the components separately:

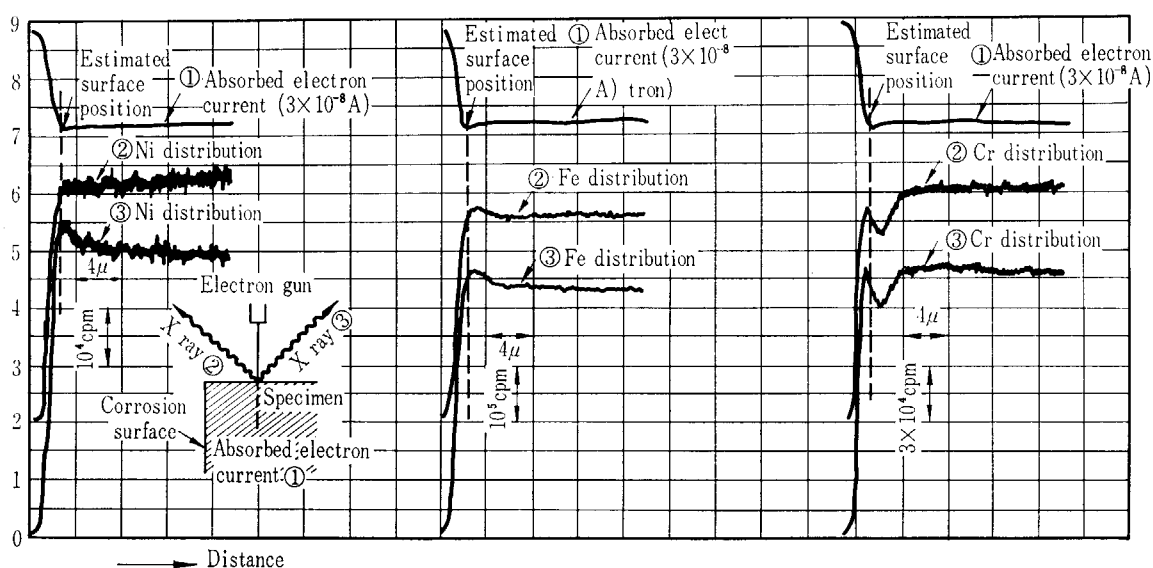


Fig. 18 X-ray microanalysis of element-distribution in type 304 stainless steel the high temperature corrosion test

- (1) Ni: No changes were observed for 304, 316 and 347 stainless steel. The inflection point in the absorption electron line (1) is probably the corrosion surface.
- (2) Cr: In every specimen, decrease in chromium was observed near the corrosion face to the depth of about 5 μ . Especially notable is the fact that at about 2 to 3 μ from the corrosion surface, there was again an increase in the chromium content. The depth agrees with the depth of the carbide layer observed with microscopic photograph and therefore the increase is probably due to the formation of chromium carbide. In other words, although the chemical activity of chromium is monotonically decreasing from the interior to the surface due to selective dissolution into sodium and the carbide formation, chromium concentration is increasing near the surface due to the reaction with carbon in the sodium.
- (3) Fe: Iron was increasing near the corrosion surface. This agrees with the region of decreasing chromium content. From this, it is thought that due to the decrease in chromium, iron with lower solubility in sodium must be increasing its concentration relatively.

The results reported here are the results of preliminary test and thus one of the interesting objectives for a future test is a study of the chromium behavior.

5. Development of supplementary techniques

5.1 Sodium metal and chemical analysis

(1) Sodium metal

The sodium used in the experiment was produced by Showa Denko Co.. Metallic sodium from Castner's method was allowed to settle for four hours at 130°C and then underwent forced filtration by a stainless steel mesh (400 mesh) at 110°C or by a 5~10 μ sintered filter. Then the sodium was purified further by a cold trap at 110~130°C. The specification is shown in TABLE 6. Calcium content was particularly low because the Castner's method was used. Carbon content was too high but was found to be reduced to about 30 ppm by another filtration.

TABLE 6 Chemical analysis of reactor grade sodium (Showa Denko Co.)

Na	99.9% <	Mg	12~17 ppm
K	2200 ppm >	B	0.01 ppm >
Ca	3~4 ppm	Cl	10~14 ppm
Si	1~2 ppm	O	12 ppm >
Al	3~5 ppm	C	70~110 ppm*
Fe	5~7 ppm		* Analyzed by the authors

(2) Oxygen analysis

Karl Fischer method⁷⁾ was employed. After the separation of sodium as an amalgam, the water yielded by the addition of acid and alcohol to the remaining oxide, was determined by Karl Fischer reagent.

This method has the same problems as the amalgamation method⁸⁾ (Pepkowitz-Judd method) and the reagent blank is also a problem. But the advantage of this method is that there is no need for the complete separation the amalgam.

The sampling system consisted of an extrusion cylinder and a cut-off device shown in Fig. 19. The extrusion cylinder was set in a tank containing method sodium and after filling the cylinder with sodium by suction, the cylinder was attached to the cut-off device. The sodium sample was forced out by a rod, and a knife was operated to cut-off the extruded sodium which was dropped into a sample carrier to be taken to the analytical devices.

(3) Carbon analysis

As the analytical method for elemental carbon, the wet-combustion method⁹⁾ using strong phosphoric acid and potassium dichromate was employed.¹⁰⁾ After dissolution with steam an excess of aqueous solution of phosphoric acid was added to the sodium sample. After vaporization drying, the sample was transferred to the analytical instrument and was boiled for about 5 minutes under oxygen gas purge after an addition of oxidizer. CO₂, after being trapped by a liquid nitrogen cooled trap, was measured by mercury manometer pressure gage or gas chromatograph.

As a method for the dissolution of sodium sample, blowing CO₂ against the metal and dissolving the powder produced was considered. But in this case, the analyzed quantity of carbon became abnormally large and extreme scatter in the data was observed. Therefore, even for the analysis of elemental carbon, the contamination of the sample by air must be avoided.

Beside using the sampling device noted previously, the samples for carbon analysis were

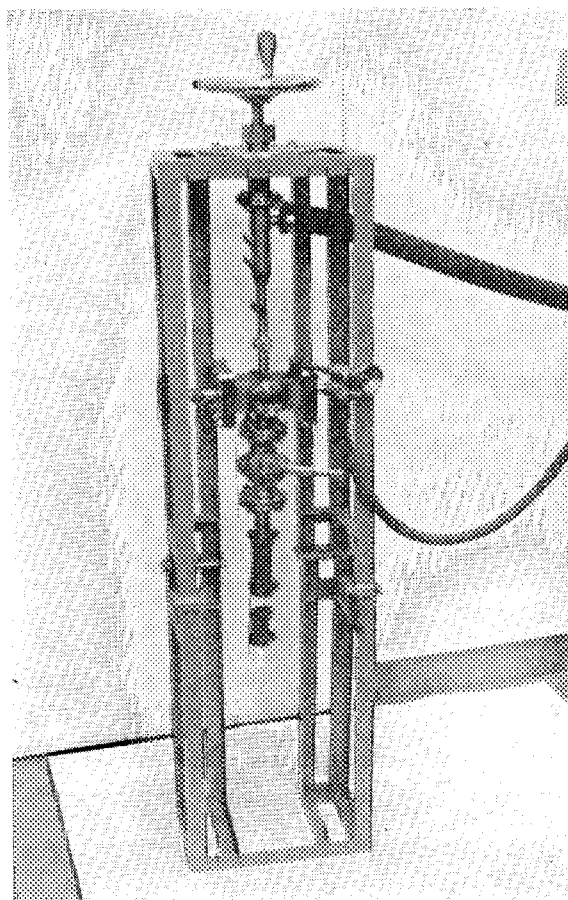


Fig. 19 Sodium sampler for chemical analysis

TABLE 7 Determination of impurities in sodium

	Carbon			Fe*	Ni**	Cr***
	No. of samples analysed	Average content ppm	Standard deviation ppm	Average content, ppm		
1. Na used in the Purification test loop	5*	75	7	7	4	0.1
2. Na used in the Mother loop	5*	73	8	3	1	0.1
3. Na used in the Mother loop	5**	79	3			
4. Na as received	6*	93	11	1	1	0.1
5. Na after filtration by 5 mesh	4*	55	6			

Weight of each sample : * 0.3~0.5 g, ** 3 g and *** 5 g.

also taken by breaking the tip of vacuum sealed glass tube inside the tank of melted sodium. After removal, the sodium solidified and then the glass tube was broken and a sodium sample was cut-off and analyzed.

When 3 gr of sodium sample, after being dissolved into an uniform solution was divided into 5 samples and separately analyzed, the data agreed well, for example, 79 ± 3 ppm (No. 3 in TABLE 7). But when samples of 0.5 gr each were taken and analyzed individually, very scattered results were obtained as shown in TABLE 7. At the present time, this is thought to be due to the fact that the carbon is not in a simple solution but is inhomogeneously distri-

buted due to the formation of a heterogeneous phase like suspension. When the filtration with a $5\ \mu$ filter is repeated, the carbon decreased. (c. f. TABLE 7)

This method can only be used with elemental carbon. But since presence of carbide, cyanide etc. is possible, an improved analytical method is necessary. Use of gas chromatograph to catch all generated carbon compound gases is being tried out.

(4) Other impurities

At the present time, Fe, Ni and Cr, in connection with the sodium system stainless steel, are being checked. After sampling in a manner similar to that for carbon, a part of the sample was cut-off and analyzed but there was a large scatter in the data. For example, results of 9, 24 and 13 ppm were obtained for Ni and an inhomogeneous distribution as in the case of carbon is suspected. The content of Cr was negligible. The mean values are presented in TABLE 7.

5.2 Impurity detection

For determining the quantity of impurity in sodium inside the loop, a plugging indicator is useful. The plugging indicator we employed is the type shown in Fig. 20. There are 17 holes

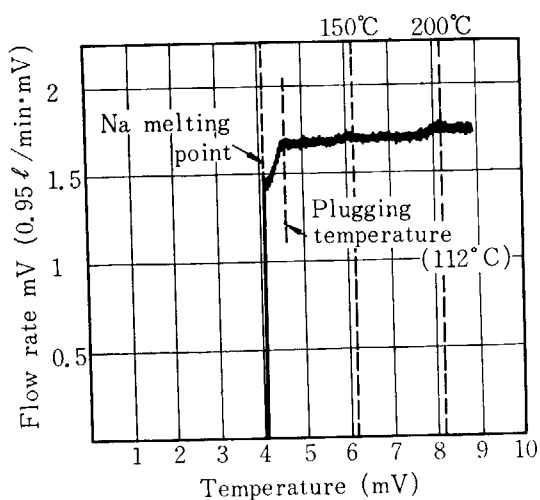


Fig. 20 Plugging indicator

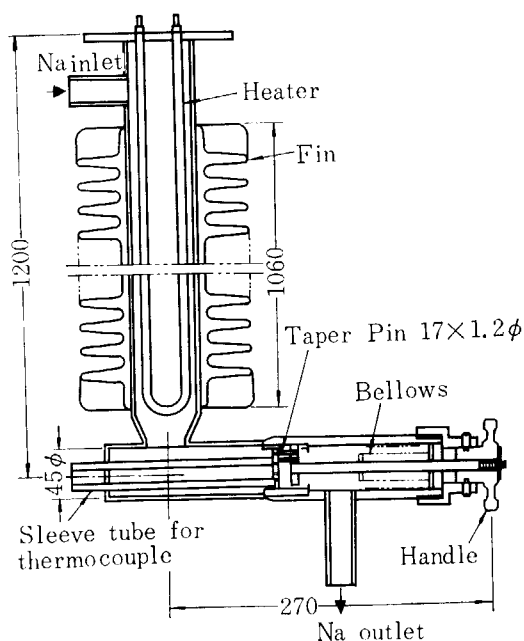


Fig. 21 X-Y recording of plugging temperature

of 1.2 mm diameter on the orifice plate of 4.5 mm thick. When, with 0.7 mm diameter pins completely inserted in the holes, if the flow rate is adjusted to 2~3 l/min and the cooling rate to 0.3°C/min, it is possible to measure down to a plugging temperature of 110°C. The required time for one measurement is about 30 min. An example is shown in Fig. 21. At higher plugging temperatures, the pins are removed to an appropriate position to facilitate measurement.

To determine, from the plugging temperature and the concentration of the impurities, the nature of the impurities and the solubility curve for the particular impurities need to be known. For oxygen and hydrogen, considered to be the main impurities, the solubility curves shown in Fig. 10 are recommended at the present time, as the most accurate curves.

With the above mentioned operation conditions, if it is desired to determine the plugging temperature with an accuracy of 1°C or 1 ppm, the following estimation can be carried out. For the temperature change of 1°C, 6~10 l of sodium will pass through the orifice and thus it must be possible to detect the effect of deposition of impurity (assumed as Na_2O) equivalent to 1 ppm ×

1/2 or 2.7~4.5 mg. This is probably sufficient to change the flow rate by a detectable amount. From the clearness of the kink in the curve of Fig. 21, it is probably possible to determine the impurities to the above accuracy.

The reason for the ability to obtain such a high accuracy determination is that the flow rate was low and the cooling was gradual, thus preventing the earlier deposition (at other region than the orifice). For 15~20 cm before the orifice, there was no cooling provided and there was an insulation to provide an uniform temperature sodium to the orifice. It is possible to improve further sensitivity of the instrument.

5.3 Level measurement

For the measurement of the liquid levels in the expansion tank and in the storage tank, induction-coil type level meter was employed. The reason for the selection of this type is that this type, except for the problem of heat-resistance, has none of the problems of the resistivity type, such as a wetting phenomena and a temperature coefficient of resistivity. And furthermore, this type can easily achieve an accuracy of ± 10 mm. As for the heat-resistance, the actual operating temperature was maximum of 500°C and thus sufficient heat-resistance was obtained by use of nickel coil insulated by borated glass fiber.

The probe consists of the excitation coil between the two detection coils. The coils, wound around ceramic core having soft steel rod in the center, are covered by a 1.5 m length type 304 stainless steel tube of 20 mm O.D.. The guide tube, welded on to the tank, has an inside diameter of 26 mm and an outside diameter of 32 mm. Using 5 kilocycle source, a manual operation resulted in a reading accurate to ± 3 mm. Since the thermal expansion at 500°C is about 1%, by considering that the upper part is cooler, the total error should be within ± 10 mm.

There was one experience that caused a problem, due to the thermal distortion of the guide tube, the movement of the probe became difficult. But this problem was solved by reducing the diameter of the upper part of the probe to 15 mm except for the coil region.

5.4 Pressure measurement

For the measurement of high temperature sodium pressure, an experimental model of a NaK (Na -78 w/o K) filled Bourdon-tube type pressure gage was made by Nagano Keiki Seisakusho Ltd. and Hitachi Ltd. by our request. Since the details will be in a separate report, a summary will be presented here.

All parts are made of type 316 stainless steel. The initial model has the highest operational temperature of 600°C, the pressure range of $-1 \sim +2$ kg/cm²g. The outside diameter is 145 mm and the effective diameter of the diaphragm is 80 mm. The pressure transfer tube has 2 mm and 3 mm internal and external diameter respectively, and the tube is in a 6 mm guard tube. A model with the transfer tube of 20 m has been made and was found to have the accuracy of ± 0.03 kg/cm². At the present time, the endurance test of the gage is under way. A photograph of the external appearance of the gage before attaching to the recorder is shown in Fig. 22.

5.5 Flow rate measurement

At the present time, five permanent magnet flowmeters and one electro-magnetic flowmeter are being used. With the magnetic field strength of 2000 to 4000 gauss, sufficient sensitivity for the flow rate of 1~20 l/min is obtained.

The meters were calibrated by allowing the sodium in the Mother loop expansion tank to flow

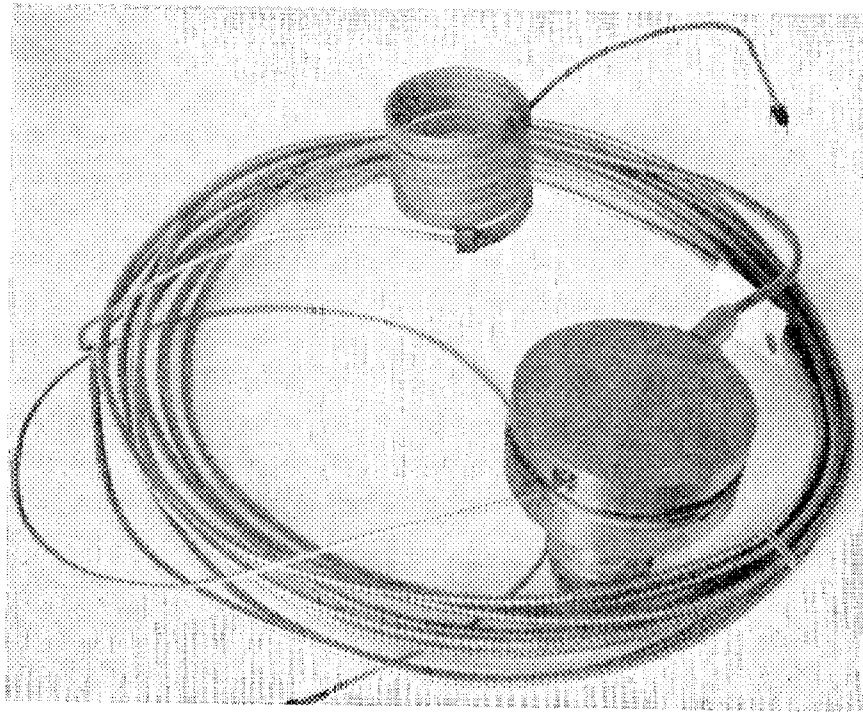


Fig. 22 Pressure gauge before attaching to the recorder

by gravity into the storage tank and estimating the volume flow by the level change. The accuracy of the measurement is about 10%. At the present time there is a lack of data on such things as the temperature coefficient. In a near future, a calibration bypass with its own measurement tank will be built and further experiments will be carried out.

5.6 Preheating and temperature measurement

Since the melting point of sodium is 98°C , the system requires preheating prior to sodium charging. It is quite important to design a simple but flexible preheating system in order to make the sodium loop operation easy. In a small loop of less than 3 inches like our test loop, the preheating was by electrical resistance, flexible sheathed heater.

The heater is MgO insulated, Nichrome electrical resistance wire thinly clad with type 304 stainless steel tube of 1.6~4.8 mm O.D.. We used mainly 2.3 and 3.2 mm O.D. heaters. The heaters are wrapped directly around the outside of the pipings and components requiring preheating and held in place by wire. The heaters are then covered with Thermon (heat conductive cement) to improve thermal contact between the heaters and the heated pipings and components to prevent local heating. Then the pipings and components with their heaters are covered with the thermal insulation of 60~100 mm thick as may be appropriate for a particular sodium temperature. At the present time, magnesium silicate type insulator and vermiculite are used as the insulating material. To prolong the life of the heaters, they are operated at a low power density level of 3~5 W/cm^2 .

The total heater power is determined by the heat capacity of the structural and the insulating materials, heat loss surface area, required temperature and the desired time for heating. In our case, the system was designed to reach 250°C in about 30 min, for example, for 1" and 3" pipings, power density was about 200 W/m and 600 W/m respectively.

In a test loop, there are occasions when the purpose of the experiment requires change in sodium flow patterns. Furthermore, in many cases, it is desirable to be able to regulate the temperature distribution freely. In some parts, sodium may become stagnant and in such a case, the

preheaters will be used again. In this case, unlike in the case of a system start-up with empty pipings, lower wattage will be required. Thus the outlet voltage of the electrical transformer must be controlled automatically or manually. In some cases, it may be sufficient to combine the heaters either in series or parallel, or to use delta or Y connection. For ease of operation, a unit length of the heaters is 2~4 m. To have two heaters in parallel also serves as an insurance against an accident. (If one heater is disabled, the other heater is used with twice the wattage.)

For the preheating of components with air cooled fins, gas burner heating is usually considered. But this has the following disadvantages:

(a) There is danger of an explosion. (b) In case of a miss-operation, there can be a local superheating. Uniform heating is difficult. (c) If the flame is accidentally blown out, a rapid cooling may result by gas blowing. These freeze-melt cycle causes of component failure.

Therefore, we wrapped flexible sheathed heaters on top of the fins. An example is shown on Fig. 11. The preheating may easily be accomplished by closing the duct. The temperature control is also quite simple.

For the loops of sizes larger than 3", preheating by A. C. induction heating is considered a superior method. Some preliminary experiments were carried out (c. f. our later report), but for the preheating of the small pipes and the small components the flexible sheathed heater was found to be superior on the following points:

- (1) Unlike induction heating, there is no problem due to the shapes of the components and the calculations are quite simple.
- (2) The installation is simple and the heater can be compact. Since austenitic stainless steel is non-magnetic, a ferritic steel mantle is necessary for the induction heating, resulting in larger pipings.
- (3) Even for a complex pipings, it is possible to design a flexible heating system. (See above.)

The temperature measurements were all by the glass fiber insulated chromel-alumel junction thermocouples. Since at least one measurement in 1 m is necessary, the method of attachment must be a simple one. The method employed was to hold down the junction welded head with a stainless steel plate of 15mm×15mm and 0.3mm thick, around which was wrapped by a steel wire.

For an accurate measurement of the sodium temperature, the junction was set in a stainless steel tube of about 4 mm I.D. inserted into the sodium stream and welded on to the pipe.

5.7 Recleaning of components

For repair and reconstruction of the sodium components, it is necessary to remove the sodium attached to the components. The methods employed at present are dry steam cleaning for the large components and alcohol cleaning for the small components. These operation are quite dangerous and the resulting phenomena are complex. Thus sufficient basic knowledges are vital for these operations.

In the case of alcohol cleaning, one must be careful of the purity, especially water content of the alcohol. There may be a danger of an explosion if an industrial grade is used. If the alcohol contains considerable amount of organic impurities, the surface of the components will be contaminated. The rate of reaction is slower for the alcohol of larger molecular weight. Therefore, using ordinary ethanol or a mixture of ethanol and butanol, etc., the temperature is regulated to obtain an appropriate reaction rate. Propanol is somewhat more expensive.

The next factor which requires attention is the solubility of the alcoholate, resulting from the reaction, in the alcohol. The result of the solubility measurement is shown in TABLE 8. When

TABLE 8 Solubility of alcoholate in its own alcohol

	15°	25°	35°	50°C
CH ₃ ONa in CH ₃ OH	27.8	28.7	29.8	31.3 w/o
C ₂ H ₅ ONa in C ₂ H ₅ OH	3.5	4.7	6.0	7.9 "
C ₄ H ₉ ONa in C ₄ H ₉ OH	0.5	0.9	1.2	1.7 "

the solubility is exceeded, there is danger due to the metallic sodium remaining under the surface crust. If an inert gas is bubbled through the alcohol, besides agitating alcohol, the hydrogen generated can be removed. When, near the end of the reaction, as the reaction becomes mild, alcohol is gradually replaced one of higher water content. In the end, a thorough cleaning with water is applied.

There are following advantages to the dry steam cleaning:

- (a) Sodium is maintained in the liquid state by the steam and the reaction rate is slow. Furthermore, the hydrogen gas generated is safely blown out by the steam.
- (b) If necessary, the reaction rate can be slowed down by dilution with an inert gas.
- (c) Steam itself is non-combustible.
- (d) Steam is economical.

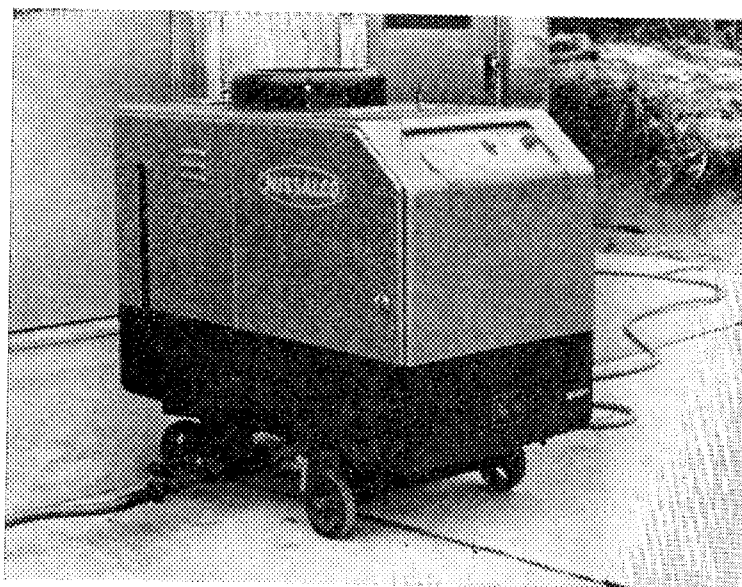


Fig. 23 Dry steam cleaner

Thus, a movable dry steam generator of the type shown in Fig. 23 is used by connecting to a component to be cleaned by a stainless steel bellow-hose and a silicone-rubber hose. The steam generator can provide steam of about 300°C, 1.5~1.7 atm with the water input of about 1 l/min. Before the cleaning operation, the component to be cleaned should be preheated. If there is a danger of an explosion, the operation is carried out remotely using a large tongues with ball joints.

5.8 Hazard protection

Some of the measures necessary for the prevention of sodium hazard will now be considered.

To prevent the leaking sodium from coming into contact with the personnel or the other components or pipings, all components and pipings must have pans or trays beneath them¹¹⁾. In case of a sodium leak accident, a natural stoppage of the accident can not be expected and thus at an appropriate time, the sodium must be dumped. The dump line, dump tank and dump valves must always be kept preheated to assure dumping in case of any emergency. The system and

TABLE 9 Endurance test of materials for burning sodium (1~2 g)

Materials	Weight (g/dm ²)	Thickness (mm)	Results (time for penetration)
Glass cloth (Al coated)	3.6		0 sec.
Cotton [canvas] (flame proof)	5.7		0
" (Al coated)	6.0		3
" (Al coated, flame proof)	6.0		5
" (water proof)	12.5		5
vinylon	4.5	0.5	5 contract
" (stainless steel foil [0.01 mm])	6.2		10 contract
" (double-ply)	12.0	2.0	15~20 carbonizing
chrome leather	5.0	0.7	0 contract
"	8.4	1.0	10 contract
"	9.0	1.2	15 contract
asbestos (Al-coated)	8.2		10
polycarbonate plate		1.5	10 burning
"		2.5	20
acrylic acid resin plate		2.5	20 softening, burning
polyester plate		1.2	30 burning
" (S. S. foil [0.02 mm])		1.5	60~120 carbonizing



Fig. 24 Protective clothing for Na fire-fighting

the surrounding part must always be kept dry and the surrounding area must be free of all water pipings. If cooling is required, air or organic coolant is used.

Various materials for the protective garments were tested. In this test, the time required for about 1~2 cc of burning sodium to burn through a test piece of garment was measured. A part of the results is shown in TABLE 9.

TABLE 10 Powder extinguisher test results

Materials		Test Result
(1) Dry Chemical	main component : sodium bicarbonate	Not effective in small quantities but was effective and did not sputter if the burning sodium was almost completely covered.* If the extinguishing material at the surface is removed, the sodium will start burning again. There is a need to guard against carbon monoxide. (* about 1 cm on the burning surface)
(2) Met-L-X (or Mytex)	m. p. : 800°C component : NaCl (damp proofed) grain size : 60~200# 50~90% 200~300# 10~50%	About the same effect as (1) but has 2~4 times the durability of (1) until the restarting of the fire.
(3) TEC	m. p. : 555±5°C component : BaCl ₂ 51% NaCl 20% KCl 29%	The effect is much less than with (1) or (2). Because the m. p. was too low, TEC flows off the sodium and the exposed sodium started to burn again.
(4) MS-3	s. p. : 160~2800°C component : (NH ₄) ₃ PO ₄ polymers H ₂ O < 1 %	When a small quantity was used it immediately started to burn, and when a large quantity was used heavy sputtering was observed.
(5) Sand		When a large quantity is used heavy sputtering resulted and gradually sodium wetted the sand surface resulting in flames and sparks.

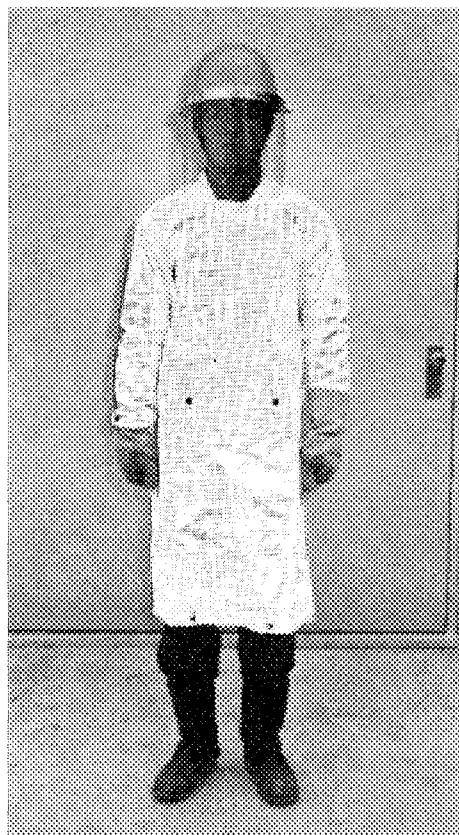


Fig. 25 Protective clothing for Na loop operation

Light weight and the best material was vinylon (polymerized polyvinyl alcohol) with an attached foil of stainless steel or nickel foil (0.01 mm thick), although thick chrome leather and aluminum coated asbestos were also acceptable. Since double-ply vinylon has the best heat-resistant property, nickel foil covered, thin double-ply vinylon with the weight of about 6 g/dm² is probably most effective.

The fire fighting garment, which resulted from these results, is shown in **Fig. 24**. On top of a flame-proofed vinylon undergarment, a flame-proofed vinylon clothing (about 0.9 kg) covered with 0.01 mm thick nickel foil is worn. This garment is designed for easy removal. It is lighter than leather or asbestos garment and is quite durable. Air is blown into the space in between the body and the garment to cool the space and this air also reduces the weight feeding of the garment.

Heavy chrome-leather is acceptable for the gloves. Boots should also be of leather. The hat was made of a nickel foil (0.02 mm thick) covered polyester with a polycarbonate plate face shield (2.5 mm thick). For smoke protection, air is blown in from the top of the hat to form an air-curtain. The face shield is not sealed and thus if unnecessary the air supply can be halted without a breathing problem. There is a muffler attached to the air line.

For ordinary sodium loop operation, flame-proofed vinylon clothing, chrome-leather gloves, leather boots, and polyester hat with a polycarbonate face shield (1.5 mm thick) are worn (c. f. **Fig. 25**).

The powder and the blanket types are considered for used as the fire extinguisher for sodium. The results of the powder extinguisher test are shown in **TABLE 10**. This test was carried out by burning about 4 kg of sodium on top of an iron tray. The best result was obtained by covering with 10 mm thick of Met-L-X (or Mytex in Japanese trade name). Drychemical can also be recommended as an economical and general purpose fire extinguisher but there is a possibility of carbon monoxide generation. TEC has too low a melting point. For small quantity of sodium, dry sand is effective but if the sand is wet, there may be some sputtering.

6. Conclusion

In this report, we have presented a summary of the results obtained in our research and development effort in test loop components and auxiliary techniques necessary as the basis of the liquid sodium technology development. Especially, the results of the development efforts in pre-heating system, purification system and instrumentations, which are necessary for the design, construction and operation of 1~3" piping test loops, were described. The so-called Mother and Daughter test loop system recommended is particularly suitable for an efficient and advanced R and D program under a well controlled impurity concentration condition.

In addition, the results of the materials corrosion tests are discussed in relation to the behavior of the impurities. The techniques were established for the chemical analysis of the impurities in sodium, recleaning of the sodium contaminated components, and hazard protection.

In the reports to follow, more detailed results of the development program will be reported.

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