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ENGINEERING REPORT 4057

SNAP I POWER CONVERSION SYSTEM MATERIALS DEVELOPMENT

PREPARED BY

NEW DEVICES LABORATORIES, TAPCO GROUP
THOMPSON RAMO WOOLDRIDGE INC.

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FOREWORD

SNAP I is the first of a family of devices to convert nuclear energy to electrical for use in space. The SNAP Systems for Nuclear Auxiliary Power - programs are sponsored by the Atomic Energy Commission; the SNAP I prime contractor is The Martin Company. SNAP I was designed to utilize a radio isotope as the energy source.

The SNAP I Power Conversion System utilizes mercury as the working fluid for a Rankine cycle. A radioisotope is used as the energy source to vaporize mercury in a boiler; turbo-machinery extracts the useful energy from the vapor and converts it into electrical energy; the exhaust vapor is condensed by rejecting the waste thermal energy to space in a condenser-radiator.

During the SNAP I Power Conversion System development, Thompson Ramo Wooldridge has been responsible for the development of the following items:

- Turbo-machinery
  - Mercury vapor turbine
  - Alternator
  - Lubricant and condensate pump
  - Mercury lubricated bearings

- Speed Control

- Condenser-Radiator

A series of eight Engineering Reports have been prepared describing Thompson Ramo Wooldridge's SNAP I Power Conversion System development program. These are as follows:

ER-4050	Systems
ER-4051	Turbine
ER-4052	Alternator
ER-4053	Pump
ER-4054	Bearings
ER-4055	Control
ER-4056	Condenser-Radiator
ER-4057	Materials

The material in this report deals specifically with the developmental history of the materials for the SNAP I Power Conversion System. This report is submitted as part of the requirements of Purchase Order OE-0101 from the Martin Company, issued under the Atomic Energy Commission prime contract AT(30-3)-217.

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1.0 SUMMARY

Because of the environmental conditions imposed on the SNAP I Power Conversion System, it was necessary to conduct a program to determine the materials to be used in fabricating the system. The materials had to be capable of operation for extended periods of time in a high temperature mercury atmosphere and also possess desired physical properties - strength, ease of fabrication and dimensional stability. The very small operating clearances resulting from miniturization of the components made it imperative that formation of any corrosion products be minimized to avoid plugging of small flow passages and interference between parts. In addition, non-metallic materials had to be evaluated to provide a reliable insulation system for the electrical components and a method for insuring that mercury did not enter the stator windings of the alternator.

Metallic materials were investigated to determine their resistance to corrosive attack by mercury and the effect of mass transfer due to temperature and concentration gradients existing in the system. Materials evaluated included the refactory metals, carbon steels, and the 300 and 400 series stainless steels. As a result of the metallic materials effort, it was concluded that the corrosion contaminants generated produce a more severe problem than the loss of structural material.

A non-metallic materials program was conducted to develop suitable insulation and sealing materials for the alternator stator. It was established that the materials and fabrication techniques develped could satisfactorily protect the stator in the SNAP I environment.

Proof that the materials used in SNAP I were satisfactory was the successful achievement of 2510 hours of full power endurance running of the complete SNAP I Ground Test System.

2.0 INTRODUCTION

The use of mercury as the working fluid for SNAP I presents a number of material problems not previously encountered. Several mercury power boilers were built just prior to 1940 for the power generating industry. Most of these units are now out of service. The temptation to compare this stationary mercury boiler experience to the SNAP I situation is very strong but can be misleading. The higher operating temperatures and higher rotating speeds coupled with the requirement for unattended, long duration operation, make the problems vastly different. The small size of the SNAP I turbomachinery package with its limited operating clearances increases the materials problem. Materials for the SNAP I power conversion system must be capable of withstanding high temperature mercury environments for long periods of time and also possess the thermal expansion, electrical conductivity, high-temperature strength, dimensional stability, and magnetic properties.

At the beginning of this program the state-of-the-art, in regard to materials to resist mercury at high temperature, was not reassuring. The data available were limited to the work of A. J. Nerad of the General Electric Company, reported in the Liquid Metals Handbook and in an Argonne National Laboratory report entitled: "Resistance of Materials to Attack by Liquid Metals." This work was limited almost entirely to low carbon steels and low chromium alloys. Type 304 SS and Type 310 SS had been tested at 1150°F, and were reported as having no structural possibility at this or higher temperatures. The low-carbon steels were reported as having good resistance to mercury corrosion up to 750°F, and limited resistance up to 1000°F.

The mechanisms of liquid metal attack have been discussed in great detail in the literature (2) (3) (7), and are believed to be as follows:

1) Simple solution.

2) Alloying between liquid metal and solid metal container.

3) Intergranular penetration and selective removal.

4) Reactions due to impurities.

5) Mass transfer

   a) Temperature gradient

   b) Concentration gradient

Simple solution and other solution-related phenomena appear to be the major mechanisms of mercury attack. Simple solution refers to the rate at which the container material is dissolved in the liquid metal. At higher temperatures the rate of solution increases, as do the solubility limits. The variation of solubility limit as a function of temperature is also believed to be the mechanism by which mass transfer occurs in a system having a temperature gradient.

Alloying between the liquid metal and the solid metal container can occur. The probability of this manner of attack can be predicted by phase diagrams, where they exist. However, the actual corrosion rate would probably depend on the diffusion rates in the solid metal and would be difficult to predict.

The grain boundaries of solid metals are generally susceptible to liquid metal corrosion. These boundaries can be dissolved away or embrittled. The so-called general corrosion sometimes appears to be a grain boundary attack, one grain deep. Selective removal is also common in liquid metal corrosion in which a solid metal alloy constituent is selectively dissolved, leaving a matrix quite different from the original alloy.

Reactions due to impurities are common in some of the alkali liquid metals. The most well-known is probably the increased corrosion which results from oxygen in a sodium or sodium-potassium system. No such effects are known in the case of mercury.

Mass transfer by temperature gradient results from solubility being a function of temperature. This type of attack can occur in any system in which solubility plays an important role, as it does in a mercury system. The container material goes into solution at a high-temperature area of the system and deposits in a cooler area due to the lower solubility at lower temperatures. The steepness of the temperature gradient also plays an important role in thermal mass transfer, and deposits are found not only in the coldest location in the system but many times in the area of greatest heat flux.

A limited SNAP I materials program began in Fiscal 1958. Two loop tests were conducted but the results were inconclusive. A decision was made in the fall of 1958 that the Martin Company would conduct high-temperature corrosion tests and Thompson Ramo Wooldridge would perform tests with boiling at 785°F and superheat temperatures of not over 900°F. This decision was based on The Martin Company's responsibility for the mercury boiler and TRW's responsibility for the mercury condenser and turbomachinery package, since it was estimated that 785°F was the highest liquid mercury temperature that would be experienced in TRW's components. It was also established that all materials cleaning would be done in accordance with specifications provided by The Martin Company.

The TRW SNAP I materials program was principally active during the last nine months of Fiscal 1959 and accomplished the testing of eight corrosion loops, a series of bi-metallic capsules, several bent reflux tubes and the evaluation of power conversion system parts from component and system tests.

3.0 LOOP TESTS

The first corrosion tests were conducted in two-phase natural circulation loops. Loop tests were originally selected because of their several advantages over other corrosion testing methods. The loop test gives conditions which reproduce, in part, the conditions expected in the final application; that is, they provide circulation of the fluid, a boiling and condensing region, liquid and vapor velocities, and a thermal gradient. The major disadvantages of loops are that they are costly and take a relatively long time to build and test.

During the early stages of this program (Fiscal 1958), a loop was designed which consisted of four sections of 0.5 inch diameter tubing joined with tube fittings. The intent was to be able to weigh the boiler and condenser sections before and after test, thereby determining the amount of attack. The general shape of the loop was a parallelogram with the boiling section in the lower end of one vertical leg and the condenser in the upper end of the other vertical leg. The lower horizontal leg was the subcooler and the upper horizontal leg the superheater.

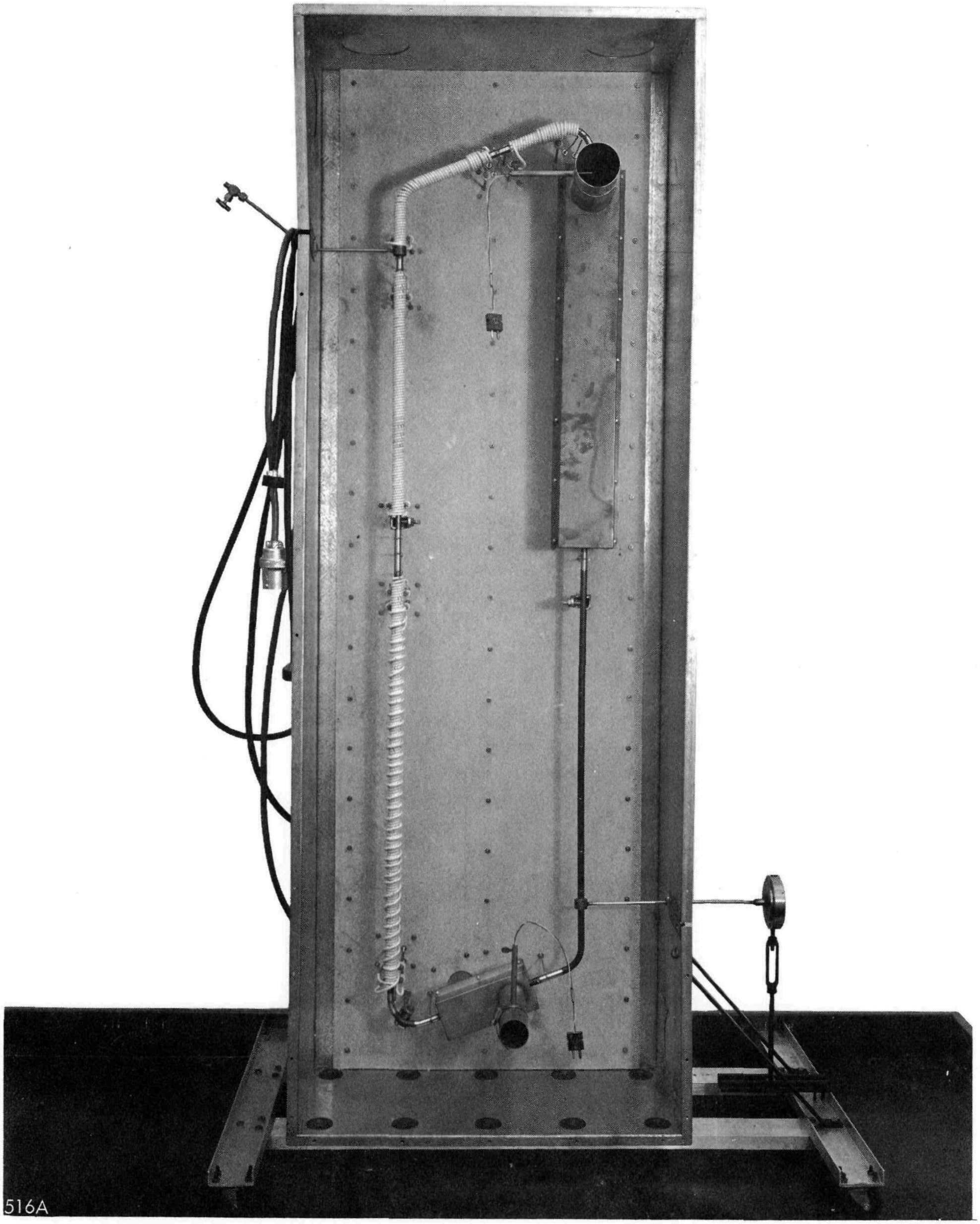
Two loops of this design were built; one was of 1010 carbon steel and the other of Type 446 stainless steel. Testing these loops indicated design and test procedure modifications required to produce more meaningful results. The tube fittings were found to be incapable of being leak tight, and the test duration of 100 hours was too short for significant corrosive attack to take place.

Figure 3-1 is a photograph of a loop of the final design installed in a metal enclosure or "can". During operation, a cover was placed on the open side and the can was filled with granulated insulating material.

The loop, as can be seen from Figure 3-1, was in the form of a parallelogram. The boiling section was the lower length of the left vertical side and the top section was the superheater. The condenser section was at the upper right hand vertical side, and the subcooler was at the bottom. The boiler heaters had a capacity over one kilowatt. However, the maximum used was 800 watts, since higher power inputs caused carry-over of liquid into the condensing section. The heat flux to the mercury was maintained at approximately 6500 Btu/ft^2-hr which resulted in a mercury circulation rate of about 12 pounds per hour and a vapor velocity of 7 feet per second.

A line heater between the boiler and superheater had a capacity of 500 watts and was used to make up heat losses. The superheater also had a capacity of 500 watts, and heated the vapor from 785°F to 850°F before it entered the condenser. The condenser consisted of an air duct surrounding a section of the loop. The duct was connected to a blower which could circulate approximately 40 cfm of air. The subcooler was similar to the condenser but smaller.

Temperatures were taken at ten locations on the loops, and were recorded intermittently on a null balance type, multiple station recorder. The temperature of each location was recorded every five minutes. The heaters on the boiler, line, and superheater were



CORROSION LOOP

FIGURE 3-1

controlled by individual on-off temperature controllers which indicated but did not record the temperature. Ammeters were located on each heater to indicate the current being used and to aid in regulating the system. The power level to each heater was controlled by an auto-transformer. The air flow to the condenser and subcooler was indicated on an inclined manometer, and was controlled manually by a valve in each air line. The mercury pressure within the loop was plotted continuously by a recorder connected to the loop through a diaphragm.

Heat losses from the first loops tested were restricted by pipe insulation fitted to the loop. Since installation and removal of this type of insulation was time consuming, it was fragile and its insulating efficiency was not high, improvements were made. The loops were next "sandwiched" between large slabs or blocks of insulating material which method, while superior to the pipe insulation, was not completely satisfactory. The blocks were difficult to handle, easily broken and expensive to replace. A third type of insulating system, devised for use on SNAP II, was used on the last loop test on SNAP I. This consisted of an insulating can shown in Figure 3-1 in which the loop was installed. This method was used repeatedly on SNAP II and was entirely satisfactory. The granulated insulating material was installed and removed by means of a vacuum cleaner in a few minutes. No heat paths, such as cracks, were encountered and the insulating material was reuseable.

Since the loops operated essentially unattended twenty-four hours a day, special safety considerations were a part of their design. The pressure recorder was equipped with an over-pressure relay that would interrupt all heater power. A second safety device consisted of an over-temperature relay so that at a preset temperature, all power was disconnected from the heaters. The loops themselves were installed in an enclosed booth which had an independent ventilating system. It was felt that the safety precaustions were sufficient and operating experience has confirmed this opinion.

Loop fabricating procedures went through a development stage during the SNAP I program. In the beginning of the program, the tube material was reamed since it was expected that a uniform diameter was required to determine the degree of mercury attack. However, it was found that the reaming operation did not permit accurate measurement of corrosion and was therefore eliminated. Annealing of the tubing before fabricating it into loops was also found to be necessary. Originally, all welds were x-rayed, but this was later omitted as being an unnecessary expense. Cleaning per The Martin Company procedures was carried out throughout the program. This was done for uniformity even though it was felt that the cleaning might have increased the corrosion in some cases.

The fabricating procedure which evolved was to cut the 0.5 inch diameter tube into the four lengths required to form the loop. Two of the lengths were then bent to form the top and bottom sections, the sections were stress relieved and welded together, complete with connections for the pressure recorder and fill line. The loop was then cleaned and leak tested with a helium mass-spectrometer leak detector.

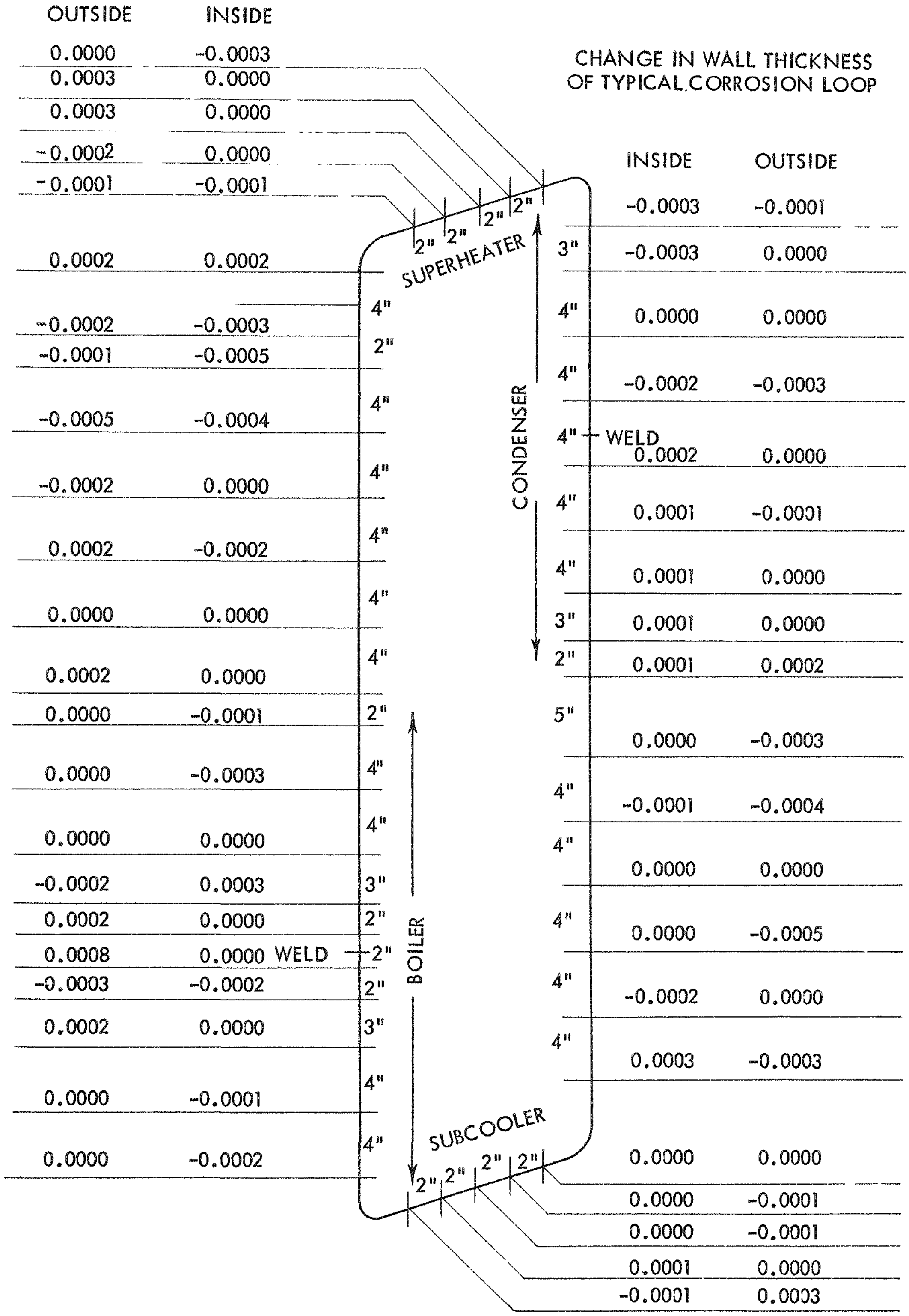
The test procedure was to first check the wall thickness of the loop at 86 points with an ultrasonic thickness tester. The loop was then installed in the can, connected to a vacuum pump and evacuated. When the pressure was below one inch of mercury, the pump was secured and the loop permitted to stand for sixteen to eighteen hours. If no pressure rise could be observed, the loop was considered to be leak tight and ready for test. The boiler, line, and superheater heaters were installed and the condenser and subcooler manifolds were positioned. Thermocouples were attached to the loop at ten locations by spot welding. A measured quantity of mercury, previously degassed by holding under vacuum for several hours, was next added to the loop. The loop was then re-evacuated, insulated, and, after checking the controls, brought to operating conditions. The operation of the loops was automatic. The temperatures recorded continuously on the loop were: boiler inlet, boiler outlet, superheater inlet, superheater midpoint, condenser inlet, condenser midpoint, condenser outlet, subcooler inlet, subcooler midpoint and subcooler outlet. In addition, the air inlet and outlet temperatures to the condenser and subcooler were recorded.

In most cases, the test conditions for all loops tested were similar. The boiling temperature was near 785°F and the superheater temperature was 850°F. The superheat was added to be sure that no liquid was carried over into the condenser. Originally the loops were run for one hundred hours, but since this was too short a time to get measurable attack, the time was increased to five hundred hours. This also was too short and the test time was extended to one thousand hours which is considered the minimum duration that gives meaningful results at the test temperatures.

At the completion of a test run, the heaters were shut off and the loop allowed to cool to room temperature. To detect any leakage from the loop, the pressure after the test was compared with the pressure at the start of the test; no change in pressure was observed in any of the loop tests. The effects of operation were evaluated by visual examination, ultrasonic thickness gauge measurements, metallurgical examination, and chemical analyses of the mercury and deposits removed from the loop. The ultrasonic thickness measurements were made as soon as the loops were drained and dismantled. Measurements were taken at the same locations as before test. Figure 3-2 shows the change in wall thickness for a typical loop.

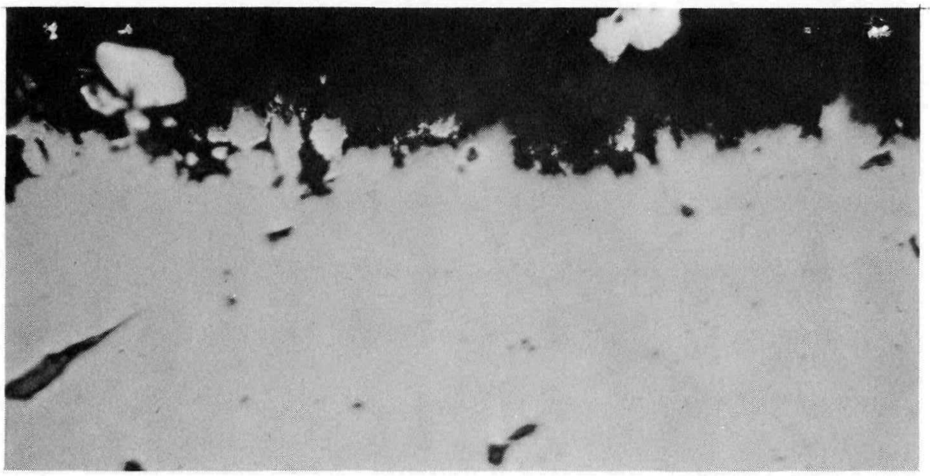
Specimens were removed from the boiler, superheater, condenser and subcooler for metallurgical examination. Typical photomicrographs of a boiler and condenser specimen are shown in Figure 3-3. The visual examination consisted of an inspection of the entire interior surface after splitting the loop, and resulted in a knowledge of the areas of attack and areas of deposit. Figure 3-4 shows the results of such a visual inspection. A spectrographic analysis was made of the mercury which was drained from the loops after test. When there were sufficient samples of deposit from the loop, they were analyzed by "wet" chemical methods.

During the course of the SNAP I program ten loops were tested. These materials, the boiling temperature of the mercury, and test duration were as follows:

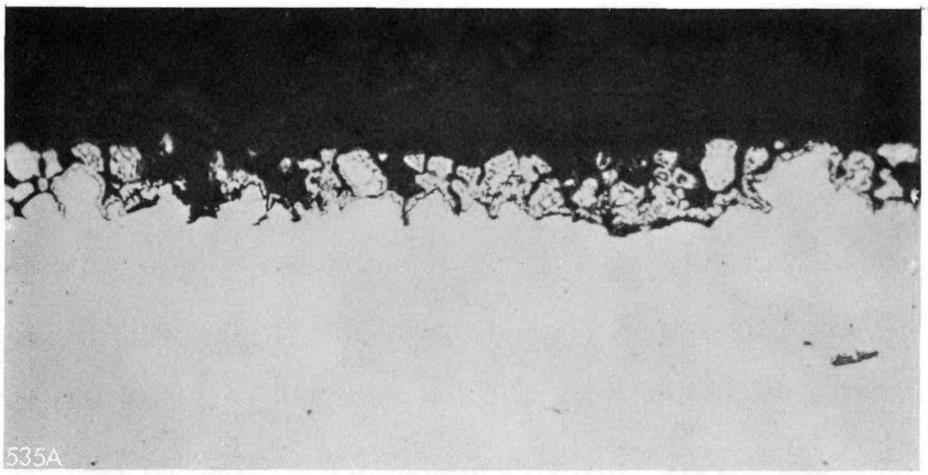


CHANGE IN WALL THICKNESS OF TYPICAL CORROSION LOOP

FIGURE 3.2



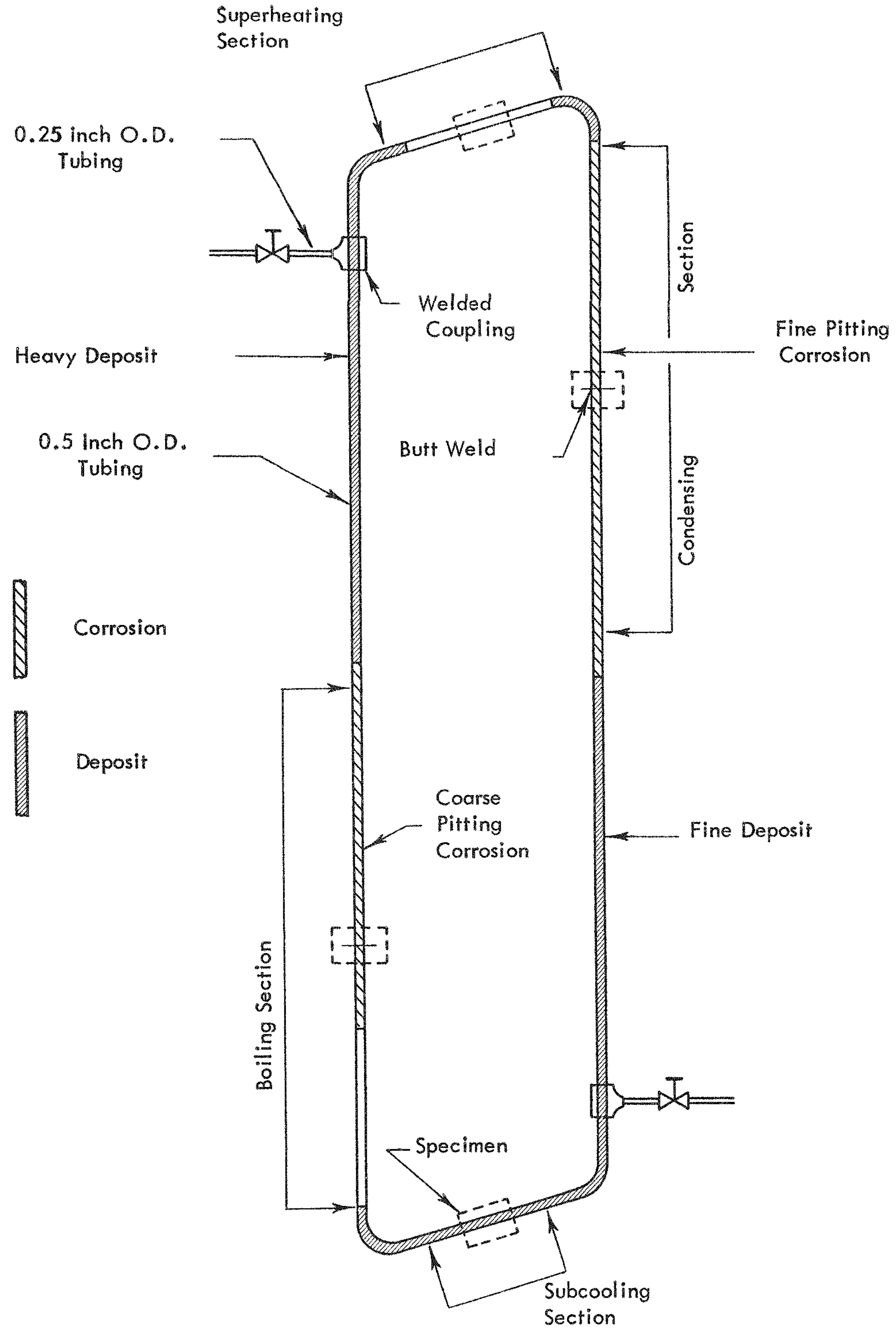
(A) MICROSTRUCTURE OF BOILER SECTION FROM TYPE 347 S.S. LOOP. UNETCHED. 500X



(B) MICROSTRUCTURE OF CONDENSER SECTION FROM TYPE 347 S.S. LOOP. UNETCHED. 500X

TYPICAL PHOTOMICROGRAPHS OF LOOP SECTIONS

FIGURE 3-3



RESULTS OF VISUAL EXAMINATION OF TYPICAL LOOP

FIGURE 3-4

Material	Boiling Temp.	Test Duration Hrs
1010 Carbon St.	750°F	100
Type 446 SS	750°F	100
Type 347 SS	723°F	500
Carpenter 20 Cb	680°F	1000
Type 316 SS	725°F	1300
Type 410 SS	784°F	1000
PH 15-7 Mo	765°F	1000
AM-350	765°F	1000
Type 446 SS	1000°F	1000
Titanium	787°F	1000

Carbon Steel 1010 and Type 446 Loops

The first two loops (1010 and Type 446) were not of the welded type and no attempt was made to measure leakage out of or into the loops. The test periods were short and no corrosion could be observed. Subsequent tests on this program and SNAP II indicated that no measurable corrosion should be expected with these materials at 750°F in one hundred hours. They are both in the second most resistant class of materials to mercury corrosion.

Type 347 SS Loop (9)

The Type 347 SS loop was tested for five-hundred hours. The general corrosion was low and not significant from a structural standpoint. However, contaminants found in the loop after the test indicated that the danger of plugging small passages in a power conversion system might be of major importance. The ultrasonic thickness gauge had not been received at the time of this test, and the quantitative data was limited. However, the metallurgical examination revealed an attack of nearly 0.001 inch in 500 hours. If this rate were to continue for a year, there would be a loss of about 0.017 inch of wall thickness.

Carpenter 20 Cb Loop (10)

A one-thousand hour corrosion test was conducted on a Carpenter 20 Cb steel loop. The boiling temperature during this test, 680°F, was lower than planned due to a test facility deficiency. Less corrosive attack took place in this loop than in the Tyep 347 SS loop.

However, at higher temperatures (900°F) tests have shown Type 347 SS to be more resistant to mercury corrosion than Carpenter 20 Cb.

Type 316 SS Loop (12)

A Type 316 SS corrosion loop was operated for 1300 hours at a boiling temperature of 725°F. From the beginning of the program, interest existed in Type 316 SS because of its high temperature properties and availability. This particular loop had been previously run to check the loop station instruments and controls. The loop was drained, refilled with fresh mercury and placed on test. After thirteen-hundred hours, heat transfer and control problems caused the loop to be shut down. The results of the examination of this loop indicated that no corrosion occurred. There does not appear to be a completely satisfactory explanation of this lack of attack. Other test experience with this material has indicated that even at the relatively low temperature of 725°F some attack should have occurred. The most significant item that may have contributed to this lack of attack was that this loop had received no cleaning before beginning the 1300 hour run and the tube surface may have been oxidized.

Type 410 Loop (13)

A one-thousand hour corrosion test was run on a Type 410 SS loop at 784°F. No corrosion was observed by either visual or metallurgical examination. A slight amount of black powder was found on the walls and in the mercury. The amount found is in good agreement with the results of the ultrasonic thickness readings. This was the first test in which the ultrasonic tester was used. The ultrasonic readings indicated an average general corrosion of about 50 microinches. If it is assumed that all of the corrosion occurred in the boiler and condenser, the wall thickness in these regions would have been reduced by 150 microinches. This level of corrosion would be hard to detect if it were general in nature. For comparison with the Type 347 SS loop, if this rate continued for a year, there would be a loss of about 0.0013 inch of wall thickness as compared with a loss of 0.017 inch of wall thickness in the case of the Type 347 SS.

AM-350 Loop (14)

The AM-350 stainless steel loop was tested for one-thousand hours at 765°F. No evidence of attack by mercury was observed visually. The ultrasonic thickness gauge indicated about twice the average corrosion found in the Type 410 SS loop. These results are in agreement with the bent reflux tube results which show Type 410 SS and AM-350 to be in the same general class of materials.

PH 15-7 Mo Loop (15)

The PH 15-7 Mo loop was tested at 765°F for 1000 hours. No attack was observed by visual examination and no deposit was found in the loop. Some "scum" was found on the surface of the mercury drained from the loop. The metallurgical examination of the loop did not reveal any positive evidence of surface attack. However, there was a possibility that some corrosion did occur in the boiler and condenser areas.

Type 446 SS (16)

A one-thousand hour test was conducted on a Type 446 SS loop at 1000°F. There was only a slight attack of this material at this temperature. The ultrasonic tests did not indicate any general corrosion but some black powder was found in the loop after the test. While some of the other loops showed similar corrosion rates, these other tests were at lower temperatures.

Unalloyed Titanium (17)

A loop of unalloyed titanium was corrosion tested for one thousand hours at 787°F. This material was the least resistant to mercury attack of any material tested under this program. It is the one material which would not be satisfactory from a strength consideration due to its high corrosion rate. Also, the amount of contaminants generated by the corrosion would be considerable. The high solubility of titanium in liquid mercury probably accounts for the results experienced.

These loop tests demonstrated that for the materials likely to be used in early power conversion systems, structural problems due to corrosion were not a problem. They also gave indications that care in material selection and pre-service treatment would be required to control products of corrosion.

The SNAP I loop tests also showed that the specified testing temperature of 785°F was too low to permit accurate quantitative comparison of materials in 1000 hour tests. Because the corrosion rates at this temperature are very low for materials of interest, higher temperatures are required to enable measurable attack to occur within 1000 hours.

The ultrasonic thickness measuring technique was found to be of some value. However, the readings are difficult to take accurately and must be evaluated in conjunction with other methods. One of the problems is that pitting attack is not detected by this method, unless it is very general in nature.

The low corrosion rates of all materials except titanium at temperatures near 700°F are in agreement with the bent reflux tube tests. The corrosion rates of the 300 series stainless steels increase very rapidly with temperature and no attempt should be made to judge corrosion rates at higher temperature on the basis of low temperature data.

4.0 BI-METALLIC CAPSULES (18) (19)

Concentration gradient or bi-metallic mass transfer has been reported in the literature for some time. (2) (3) (7) (20) However, the thermal mass transfer is generally much greater in magnitude and has received more attention. A paper by I. W. Taylor and A. G. Ward, (20) on bi-metallic mass transfer in lead, indicated that this effect could reach major proportions. It was realized that the SNAP I system could not be uni-metallic and that the bi-metallic effects should be investigated. Therefore, a test method was devised and a number of material combinations were investigated.

Glass test tubes were used to form sealed capsules in which two specimens were immersed in mercury. The specimens for these tests were cut from bar stock and were 0.25 inch in diameter and 0.50 inch long. The capsules were fabricated from glass test tubes 22 millimeters in diameter and 19 centimeters long, having a side arm 7 millimeters in diameter. The top of the test tube had a ground glass stopper which was sealed with glass frit. The specimens were separated by glass spacers. After cleaning, the specimens were installed, mercury was added to the capsule, and a vacuum was pulled through the side arm. The side arm was then heated and sealed. The capsules were placed in furnaces at 650°F for 500 and 1000 hours. Table 4-1 lists the material combinations tested.

The general conclusions from these tests are that at 650°F the following materials can be used in combination:

Type 347 SS with Type 420 SS
Type 347 SS with 17-4 PH
Type 347 SS with PH 15-7 Mo
Type 347 SS with Type 410 SS
Type 347 SS with Type 431 SS
Type 316 SS with 17-4 PH
Type 316 SS with PH 15-7 Mo
Type 410 SS with Type 316 SS

The following materials have shown bi-metallic effects in every combination listed in Table 4-1.

Alnico VI
Tungsten Carbide
Vascojet 1000
Titanium Carbide
18-4-1

The bi-metallic tests have been of great value in providing data for material selections, in that material combinations have been avoided which might have involved interactions.

TABLE 4-1

MATERIAL COMBINATIONS TESTED IN BI-METALLIC CAPSULE TESTS

Alnico VI with Type 347 SS

Alnico VI with Type 420 SS

Type 347 SS with Type 420 SS

Alnico VI with Type 347 SS and Type 420 SS

Type 347 SS with 17-4-PH

18-4-1 with 17-4-PH

Titanium Carbide with 17-4-PH

PH 15-7 Mo with Type 347 SS

Alnico VI with 17-4-PH

Type 410 SS Nicrobrazed to Type 410 SS

Type 347 SS with Type 410 SS

PH 15-7 Mo with Type 316 SS

Type 410 SS with Type 316 SS

17-4-PH with Type 316 SS

Tungsten Carbide with Titanium Carbide

PH 15-7 Mo with Type 316 SS

Alnico VI with Type 347 SS

Tungsten Carbide with 17-4-PH

17-4-PH with Vascojet 1000

Titanium with Alnico VI

Type 347 SS with Type 431 SS

15-15 N with Alnico VI

Type 431 SS with Tungsten Carbide

Type 431 SS with Vascojet 1000

The limitations of these tests has been that the temperature could not be increased over 675°F due to capsule design limitations. Higher temperatures have been investigated under the SNAP II program in bent reflux capsules.

5.0 BENT REFLUX TUBES

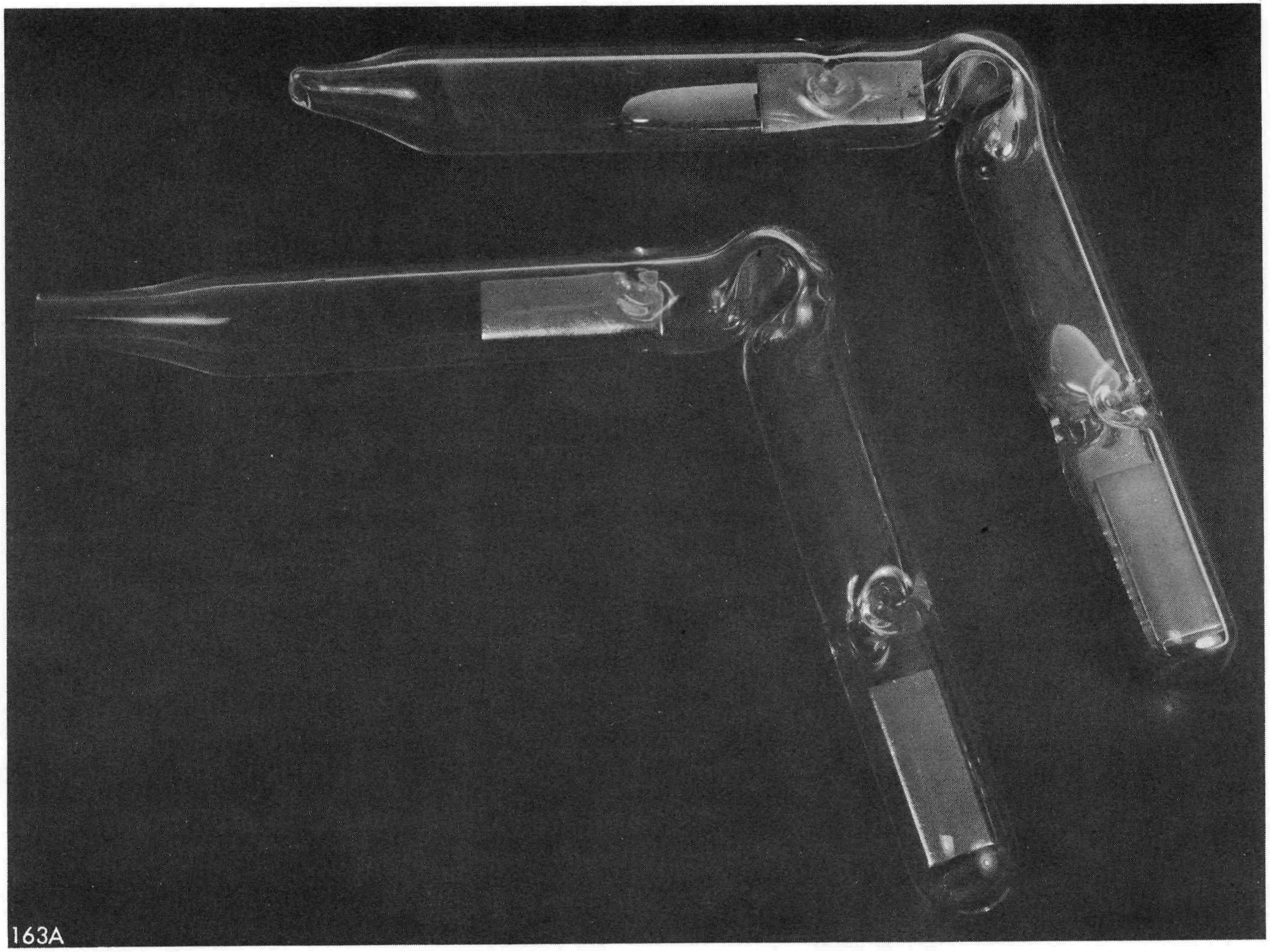
The use of capsules and other static methods of corrosion testing have certain inherent disadvantages. Some of these disadvantages are particularly pronounced in the case of corrosion testing with mercury since solution phenomenon appears to be one of the major mechanisms of attack in the case of mercury. Therefore, the rate of attack depends on the solubility of the container material at a given temperature and the amount of material in solution. If the mercury is saturated with the container material, no more attack will occur. In a capsule test, saturation may occur rapidly and further duration of testing would be both misleading and useless. Another disadvantage of capsule or static tests is the lack of circulation, since it is thought that velocity is of major importance in conducting corrosion tests. Also, temperature gradients, which reveal mass transfer, are not possible in capsule tests.

Early in the SNAP I materials program, it became obvious that the lack of mercury corrosion data would require the screening of a large number of materials. Because of the cost and time involved to conduct loop tests, a simple test device was needed to accomplish this. Such a device, the bent reflux capsule, was developed by Thompson Ramo Wooldridge and is shown in Figure 5-1. This test method has overcome some of the major objections to static capsules. The bent reflux capsules consisted of pyrex glass tubes formed into an inverted "L". A specimen was installed in the horizontal leg and heat was applied to mercury in the bottom of the vertical leg. The mercury distills from the vertical leg into the horizontal leg where it condenses and covers the specimen. As more mercury condenses, a level is reached where it overflows back into the vertical leg. In this manner the specimen is exposed continuously to freshly distilled mercury. The distilling and reflux action of these capsules eliminates the major objection to capsule tests; that is, that the mercury becomes saturated and limits additional corrosion.

The bent reflux tubes are well suited for preliminary screening of materials to determine the effects of composition, stress, and surface treatment. They also can be used to study bi-metallic corrosion, the effect of additives, and the increase in corrosion as a function of time or temperature.

The tests conducted as part of the development of the bent reflux tubes and those conducted for SNAP I are listed in Table 5-1. These tests were not extensive - only about 25 materials were screened by this technique before program termination. Under the SNAP II program over 300 bent reflux tube tests were conducted for various periods of time and at temperatures of 700°F to 1100°F. A number of additives were also investigated.

The test conditions and results of the SNAP I bent reflux tube tests are shown in Table 5-1. Those results are in good agreement with data from loop tests and similar tests from the SNAP II program. One exception is titanium, which has been shown to have very poor resistance to mercury, was only poor in these tests.



MERCURY REFLUXING TUBES

FIGURE 5-1

TABLE 5-1

RESISTANCE OF MATERIALS TO LIQUID MERCURY CORROSION AT 900°F AS TESTED IN THE BENT REFLUX TUBE

Material and Condition	Test Time	Hg Adsorbed	Weight Change	Corrosion Layer Thickness	Remarks
	Days	mg	mg	Inch	
Titanium carbide (K150A)	26	0.15	0	0	Spec. not wetted
Titanium carbide	12	0	0	0	Spec. not wetted
Tungsten carbide (CA-8WC)	12	0.2	0	0	Spec. not wetted
Ferrotic (1760°F, W.Q., 10 min. 375°F)	12	0	0.2	0	Spec. not wetted
Tungsten Carbide (K96)	12	0.1	0.1	0	Refluxaction not proper
Titanium Carbide (K162B)	12	0.4	0	0	Spec. not wetted
Columbium (cold rolled)	12	—	-0.9	0	Spec. wetted. Laminated coupon. results may be erroneous (spec. forced into tube).
Tantalum (annealed)	26	0.1	0	0	Spec. not wetted
Inconel (annealed)	12	3.7	-49.4	0	Wetted spec. some areas eaten away
Type 410-annealed (ferrite and carbides)	12	0	0	0	Slight wetting
Type 410-1800°F, W.C.	12	.05	-0.3	0.0005	Intergranular penetration
Type 410-1800°F W.Q. 1350°F Temp.	12	.05	-0.05	0.0005	Intergranular penetration
Type 410-annealed	37	0	-0.7	0	Acid cleaned
XCR valve steel (2100°F, W.Q. 14 hr. 1400°F)	12	0.9	0.1	0	Slight wetting
17-4PH (1-1/2 hr 1400°F, A.C. to 60°F 1-1/2 hr. 1050°F)	12	0	-0.8	0	Not wetted
17-4PH (Condition A)	12	0	-0.7	0.0008	Not wetted
Carpenter 42 L. E. (Cold rolled)	37	211.6	-139.5	0.01	Wetted
Carpenter 42 L. E. (annealed)	12	43.0	-32.5	0.002	Wetted
Type 304L (annealed)	12	7.2	-4.1	0.0028	Slight wetting
Type 304L	5	—	-3.0	0.0012	Acid cleaned
Type 304L	10	—	-5.4	0.001	
Type 304L	43	2.8	-12.7	0.0024	Acid cleaned
Type 304L (Stressed)	12	—	—	0.0032	
Type 347 (annealed)	12	25.2	-14.8	0.0044	Slightly wetted
Type 347	11	—	-14.0	0.006	Acid cleaned
Type 347	7	—	-11.7	0.0016	Acid cleaned
Type 347	5	—	-7.7	0.001	Acid cleaned
Type 347	43	7.2	-9.7	0.0024	Acid cleaned
Type 347 (Stressed)	12	—	—	0.002	Acid cleaned
15-7Mo (Condition A)-24 hrs. oxidation at 900°F	10	—	-0.2	0.0016	Acid cleaned
15-7Mo (Condition A)	10	—	-1.1	0.0018	Acid cleaned
15-7Mo	10	—	-0.9	0.0016	Acid cleaned
15-7Mo	5	—	0	0.0012	Acid cleaned
15-7Mo (1-1/2hr 1400°F A.C. 1/2hr. 50°F, 1-1/2 hr 1050°F)	11	—	0	0.0008	Acid cleaned
15-7Mo (Condition A)	43	5.8	-4.7	0.04	Acid cleaned
15-7Mo (Hardened)	67	1.4	-2.9	0.0005	Acid cleaned
15-7Mo (Condition A) (Stressed)	12	—	—	0.0012	Acid cleaned
Carpenter 20Cb (Annealed)	19	—	-92.5	0.0054	Acid cleaned
Carpenter 20Cb	43	92.0	-148.0	0.062	Acid cleaned
Zircalloy-1 (annealed) (2-3 % Sn)	12	424	-223	0.046	Wetted
AISI 1095 (Hot rolled-ferrite and carbides)	12	0	-0.15	0	Slight wetting
AISI 1010 (annealed)	12	0	-0.4	0	Slight wetting
18-4-1 Tool Steel (Tempered martensite and carbides)	12	0.2	-0.4	—	Not wetted, condensing action not proper
	12	0.3	0.3	0	Not wetted, same sample as above
AM350 (hardened)	12	0.6	-0.3	0	Slight wetting
Tungsten	12	0	0	0	Not wetted
Titanium (annealed)	12	30	-33.5	0.0014	Wetted 0.01" eaten away
Stellite No. 6 (as cast)	12	-0.9	-0.9	0	Wetted slightly
Thermenol (2-1/2 hr 1100°F)	12	0	0.4	0	Not wetted
Pyroceram No. 9608	12	0	0.1	—	Not wetted
Molybdenum (0.5% Ti)	73	0	0	—	Not wetted
Type 446 (annealed)	12	0.2	-0.2	0.0017	Not wetted, acid cleaned
Type 446	43	1.0	-1.4	.0029	Wetted, acid cleaned
Type 403 (1800°F, W.Q. 1350°F Temper.	43	0.5	-0.3	0	Wetted, acid cleaned
Type 403	12	0	0.2	0	Not wetted, acid cleaned
Cobalt (as cast)	12	32.5	0.1	0	Wetted completely
Chromium (as cast)	1	0.4	-0.4	—	Wetted

As indicated previously, the bent reflux tubes tested under this program were too few in number to draw any conclusions. However, the correlation of these tests with loop tests proved their value to the corrosion program.

6.0 COMPONENT PARTS EVALUATION

A major source of practical information relative to the corrosion resistance of materials is obtainable from the examination of prototype components which have been tested under operating conditions. One phase of the materials effort under the SNAP program was the metallurgical examination and chemical analysis of such components. All components which failed or showed attack were examined, and the mercury taken from tested units was analyzed chemically. Test facility components were also subjected to the same examinations.

Between the SNAP I and SNAP II programs, practically every component of the rotating unit and the test rig came under chemical or metallurgical scrutiny at some time.

In general, it can be said that the results of the PCS (Power Conversion System) component examinations supported and augmented the data obtained from capsule and loop corrosion tests. In addition, some attack was observed which resulted from velocity of the mercury vapor and entrained moisture. The opportunity to discern this type of attack was not present in the corrosion tests.

The chemical analyses have been performed by both spectrographic and "wet" chemical methods. In general, the spectrographic analyses have been too sensitive in that they have revealed trace amounts of every material with which the mercury came in contact. This is very satisfactory when working with the corrosion tests, but when trying to analyze the mercury from a complete PC (Power Conversion) system, the results are difficult to interpret. The wet chemical analyses have presented a problem in obtaining accuracy, but have been of great importance in analyzing the attack observed by visual and metallurgical examinations.

7.0 CONCLUSIONS

1. The loop and bent reflux capsule testing methods enabled an order of merit ranking of the following materials to resistance to corrosion by mercury. Beginning with the most resistant materials, they are:

    a. Tantalum, Tungsten, Molybdenum, Titanium Carbide, Tungsten Carbide.

    b. 410, 446, 17-4 PH, Columbium, 1010, 15-7 Mo, 1095, 403, AM-350.

    c. 302, 304, 316, 321, 347.

    d. Carpenter 20 Cb, Carpenter 42 LE, Inconel, Titanium, Zirconium.

2. Bi-metallic capsule testing indicated that certain combinations of materials in a mercury system would increase corrosion.

3. For the majority of engineering materials tested, mercury corrosion will not cause structural problems at the temperature used in testing. However, contamination of the system by products of mercury corrosion requires care in selection of materials.

4. Liquid metal corrosion testing techniques developed under the SNAP I program or concurrently with it, proved successful in providing mercury compatibility data.

5. Testing temperatures of at least 900°F are required with most engineering materials to obtain sufficient attack to obtain accurate quantitative data in 1000 hours.

8.0 INTRODUCTION

Nonmetallic materials are essential design components of electrical equipment. When applied to the problems of the SNAP I system, especially the alternator stator, a concentrated effort was required on the development of new materials, improvement of processing techniques, and the evolution of a completely new insulation system.

The nonmetallic materials portion of the SNAP I program was aimed at the development of an insulation system for the alternator stator of the turbo-machinery package. The requirements for this system were stringent. Specifically, the alternator stator had to be capable of continuous operation at elevated temperatures in a mercury vapor environment. The insulation system was required to provide the required dielectric strength to permit operation at the electrical design level. In addition, it was necessary to devise a means of sealing the mercury vapor within the bore to prevent detrimental effects on the winding and insulation system.

An insulation system capable of resistance to temperatures up to 550°F was established. This system maintained reasonable handling qualities which permitted its use in the fabrication of alternator stators. The capability of the developed insulation system to operate satisfactorily in the mercury vapor environment was demonstrated in autoclave, component, and system endurance tests.

The most difficult problem was the development of techniques to realize an alternator bore seal which was leak tight at the operating temperature of the system. This was accomplished by using materials capable of exposure to high temperatures, and resistant to the effects of liquid and gaseous mercury. Such techniques had to be developed from conception as they did not exist in a fashion of merely utilizing presently available materials.

Among the problems foreseen in the development of the SNAP I power conversion system were the selection and application of nonmetallic materials. The need for a nonmetallic materials program was emphasized by the lack of available information describing the behavior of this type of material. It was known that high temperature resistant materials might not perform well in a liquid metal environment. The nonmetallic materials requirements of the SNAP I program were generally confined to those associated with the alternator stator. Though two distinct problem areas existed, the insulation system and the bore seal, the materials evaluation program for both requirements were conducted together. In order to attain the necessary electrical efficiency and reliability required for long-duration operations, the materials for both applications were required to be good electrical insulators and compatible with mercury.

Initially, the insulation system was not considered a serious problem since the design specification called for a mercury cooled stator capable of operation at a 450°F maximum hot spot temperature. This would have been within the capability of commercially available class H insulating materials. In addition, the bore seal problem appeared to be solvable through the application of filled class H resins as bore sealing materials. The materials problems were later increased by eliminating stator cooling in order to improve system performance and reliability. The stator was then required to operate at winding temperatures up to 550°F. It was found that information concerning the stability of nonmetallic materials to long-term exposure to these temperatures was not available. Also completely absent were data on mercury compatibility.

A study phase of nonmetallic materials was set up as the initial part of the program. From this, a materials evaluation program evolved that moved into a field where new techniques and new processes had to be developed in order to utilize available and promising materials. In some cases, new materials had to be developed in order to find solutions to the problems. Thus, it became necessary to advance the state-of-the-art in order to develop the electrical insulation system required for the SNAP I program. In addition, the bore seal problem had to be solved in order to insure an efficient, reliable power conversion system. This was accomplished through as extensive a test program as was possible with the limited time and funding available.

9.0 SELECTION OF MATERIALS

Early in the investigation, it was evident that materials were available which were potentially useful on the SNAP I program. However, in most cases, the properties of these materials in the environment peculiar to SNAP I had not been evaluated. In order to intelligently select the required materials, the problem areas in the system had to be defined, and a test program instituted to aid selection of materials to suit these areas.

Silver was selected as the stator winding material on the basis of weight and volume required, and electrical performance as a function of temperature. The presence of silver, whose resistance to mercury corrosion is low, emphasized the need for a seal to prevent mercury from entering the stator windings. Even if the silver were absent, a seal would still be required as mercury vapor, in the presence of ionizing radiation, could short-circuit the windings.

The early evaluation program was aimed at developing a nonporous potting compound which would serve to insulate and support the windings, and would at the same time present a barrier to mercury penetration. As the temperature requirements were raised, the emphasis shifted to inorganic, porous potting materials which could be coated with a nonporous barrier coating.

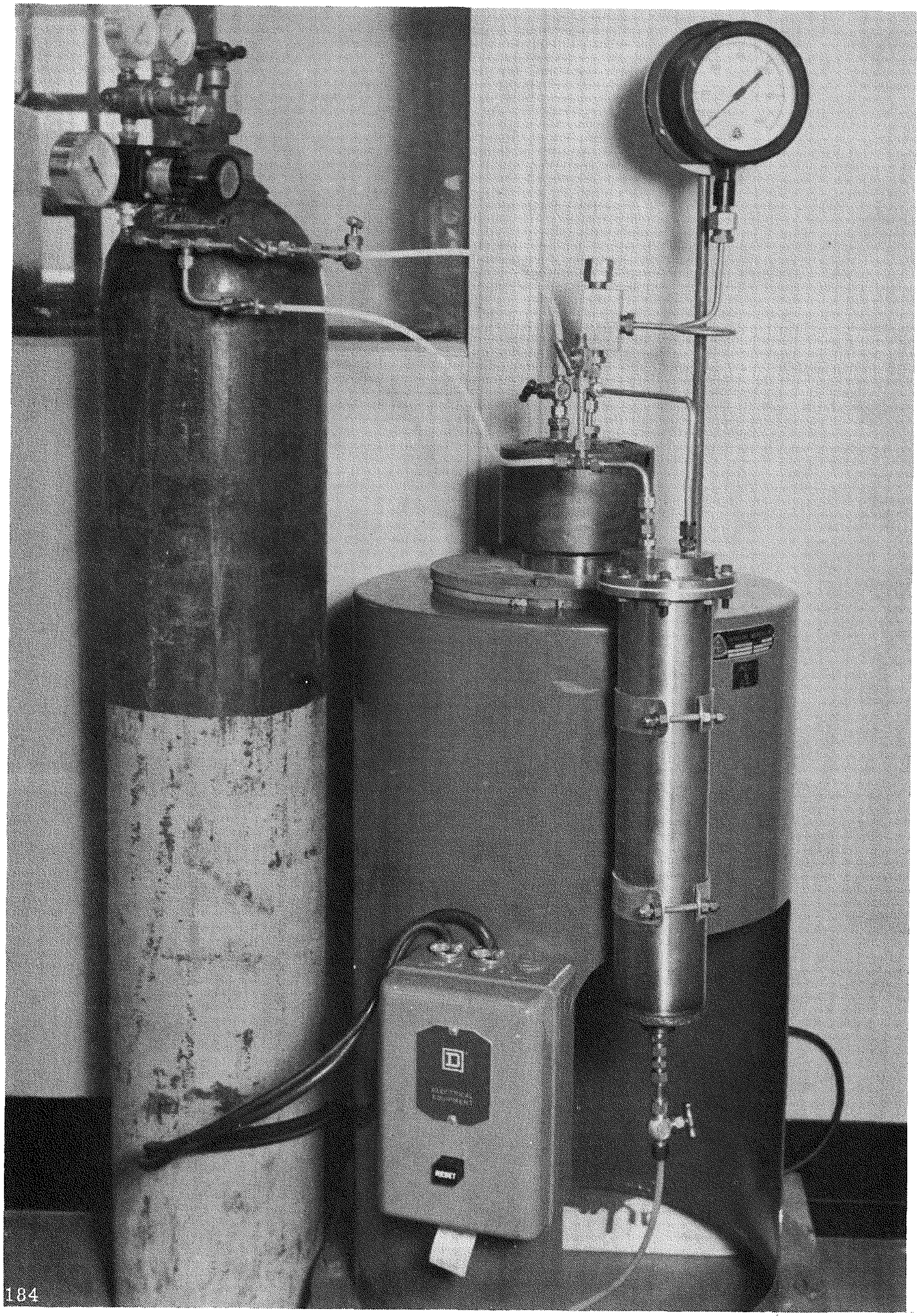
It was felt that the following tests would supply the necessary data:

1. Autoclave test - Exposure to mercury vapor at elevated temperatures.
2. Autoclave test - Exposure to mercury liquid at elevated temperatures.
3. Autoclave test - Change in resistance of coated wire coils after exposure to mercury.
4. Stability of materials after long duration exposure to high temperatures and temperature cycling in air.
5. Voltage breakdown in air.

Mercury compatibility testing was conducted in a one-liter, nonagitated pressure vessel shown in Figure 9-1.

The vessel was constructed entirely of type 316 stainless steel. The top of the autoclave was supplied with a pressure gauge, manometer, vent connection, and thermocouple well. The signal from the thermocouple was fed to a pyrometer controller which supplied power to electrical strip heaters clamped around the sides of the vessel to obtain controlled heating.

To conduct a normal test run, mercury was poured into the bottom of the autoclave to a depth of 4-6 inches. The samples to be tested were fastened to the thermocouple well with stainless steel wire in such positions as to expose one sample to mercury liquid and the other to the vapor space above the liquid.



MATERIALS COMPATIBILITY TEST TANK

FIGURE 9-1

The autoclave was bolted shut and the heaters turned on. During the heating period, the vent line was opened to prevent pressure buildup, and to permit mercury vapor to displace the air in the vapor space. All runs were made at temperatures below the normal boiling point of the mercury. Upon reaching the required operating temperature, the vent valve was closed for the duration of the run.

Upon completion of the required test duration, the autoclave was cooled, and the samples removed. The test specimens and samples of the mercury taken before and after the test were examined by spectographic, chemical, and metallurgical techniques.

10.0 TEST PROGRAM

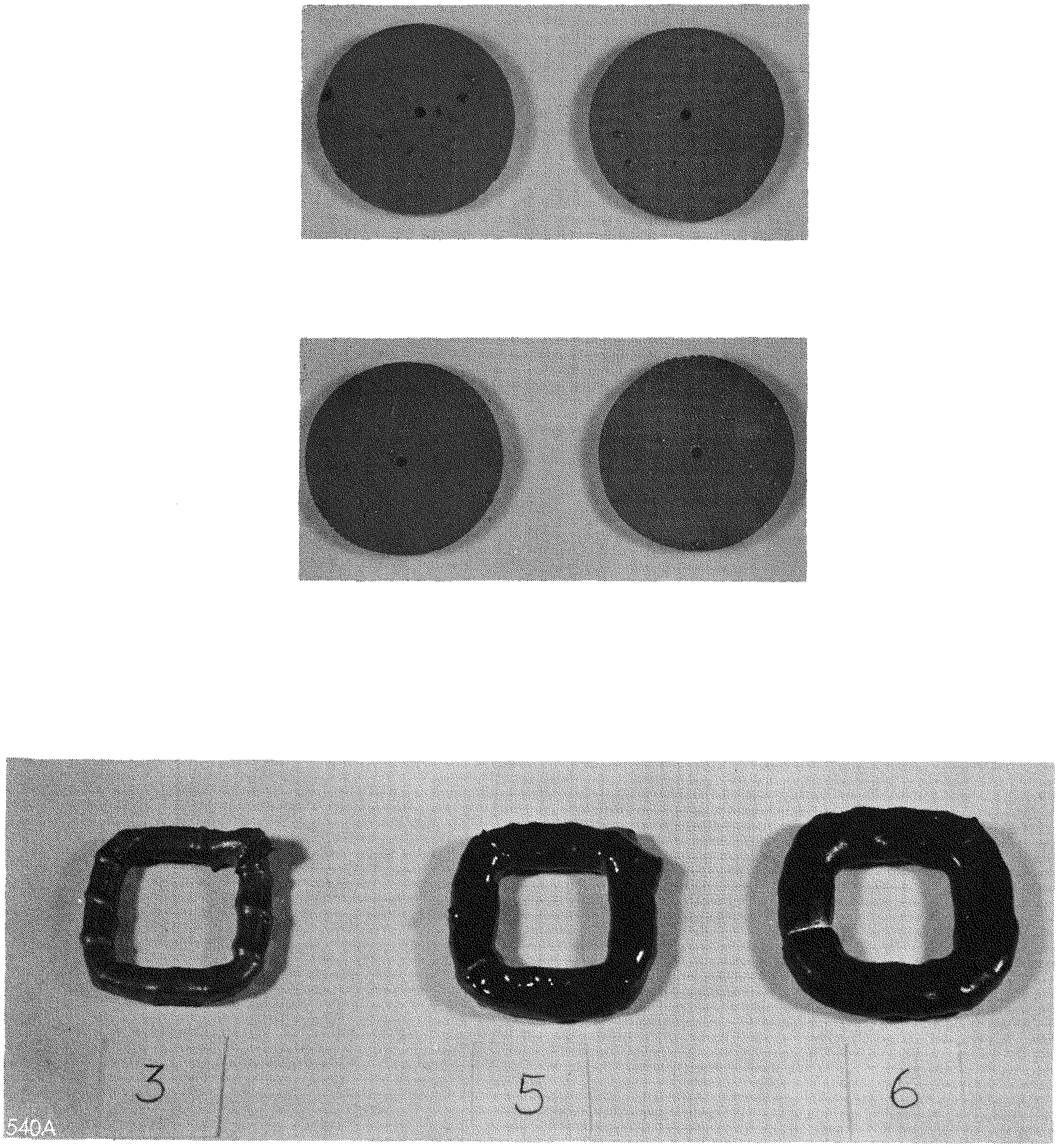
It was proposed to investigate the problem areas of compatibility, sealing, dimensional stability and mechanical property deterioration. Autoclave testing was the selected method for evaluating nonmetallic materials since it was considered to be the simplest method to determine general compatibility with mercury at high temperatures either in a vapor or liquid state. Also, this type of testing permitted flexibility in the program in that several conditions of temperature, pressure and test duration could be incorporated when screening materials. In most cases, emphasis was placed on exposing prospectively useful materials to mercury at 500-600°F. This served the double purpose of determining at least qualitatively the thermal stability and mercury compatibility of the materials in question.

In general, two types of samples were tested. Either discs of the pure materials, or double-glass-served copper coils encapsulated in the material were used. Figure 10-1 shows typical test specimens. After exposure to the desired conditions, the samples were examined visually for physical and dimensional changes. Impurities in the mercury were determined by spectographic methods, and chemical and metallurgical examinations of the specimens were made. A summary of the test program and the results obtained is given in Table 10-1.

Those materials which showed promise during the test program were then evaluated with reference to their handling properties, ease of fabrication, and other properties which reflected their ability to be incorporated physically into the desired insulation/sealing system.

The investigation of a curing process for potting the alternator stator with Pyroceram was in effect at the termination of the SNAP I program since much success had been experienced with Pyroceram as an impregnant and coating. It was believed that processing techniques could be obtained whereby small defects such as bubbles and cracks in the bore seal could be minimized and possibly eliminated. The ultimate goal was a leak-tight bore seal which would guarantee a stator completely impervious to mercury attack.

These further studies with Pyroceram were to have been devoted to the investigation of application techniques and combinations of curing temperatures and times.



TEST SPECIMENS

FIGURE 10-1

TABLE 10-1

	Material Tested	Test	Result
1.	Allan P-1 cement and Allan PBX cement	Cured per instructions.	Extremely porous--not evaluated further.
2.	Emerson and Cuming Eccofoam HiK and Eccofoam	Potted coils in the compounds and cured.	Extremely porous--no further evaluation.
3.	"Special High Temperature" potting compound	Potted coils submerged in liquid mercury for 6 hours at 600°F.	Complete saturation with mercury. Hg between windings and coating copper wires.
4.	"Special high temp" potting compound sealed with "Special High Temperature" resin	Coils were potted in the compound was then coated with the resin. Exposed to Hg liquid for 6 hours at 600°F.	Saturation of compound and coil with Hg. Hg on surface of copper wire.
5.	Silicone varnish (50% solids)	Coils impregnated with varnish were exposed to mercury at 500°F.	No penetration evident.
6.	Silicone varnish filled with mica flour	Material exposed to mercury at 500°F.	Appeared very porous--no cracks evident.
7.	Silicone varnish filled with magnesium oxide	Exposed to mercury at 500°F.	Very porous--many cracks in surface.
8.	Shell Chemical Co. Epon 828 resin catalyzed with PMDA	Exposed to 500°F temperatures.	Failed due to temperature.
9.	Shell Chemical Co. Epon 828 resin catalyzed with PMDA, mixed with THFA	Exposed to 500°F temperatures.	Failed due to temperature.
10.	100% silicone resin, Dow Corning DC-7521	Exposed to mercury for 7-1/2 hours at 500°F.	No penetration evident.
11.	D7-7521 resin filled with 160-180 mesh zirconium orthosilicate	Specimens of material were exposed to both Hg liquid and Hg vapor for 112 hours at 600°F.	Visual observation under 40 x magnification showed no apparent penetration.
12.	DC-7521 resin filled with 160-180 mesh zirconium orthosilicate	Coils were potted in the compound and exposed to Hg vapor and liquid at 600°F 96 hours.	No evidence of cracking or penetration seen under 40 x magnification.
13.	Corning Glass Works Pyroceram No. 95 solder glass	4 coils, impregnated and coated with the glass were exposed to Hg vapor for 100 hours at 500°F and 2 psia. 2 coils were "glassy and two were devitrified.	40 x examination showed no Hg in the Pyroceram. Chemical analysis of successive layers of the glass surface shows no mercury. Cracks in the glass permitted Hg to reach the copper.
14.	Pyroceram No. 95 solder glass	2 coils, impregnated and potted, were floated on liquid mercury. The autoclave was cycled by heating to 600°F for 8 hrs each day & cooled overnight. Total exposure-100 hrs. Pressure was 1 atmosphere throughout.	Radial cracks found in the glass. Mercury found in coils was assumed to have penetrated through the cracks rather than the glass.
15.	Pyroceram No. 95 solder glass	4 coils, 2 "glassy" & 2 devitrified. Cyclic test in air. 100 cycles were conducted by heating to 600°F, holding for 1/2 hrs, the cooling to room temperature & holding there for 1/2 hr to complete the cycle. Resistance of coils measured at high and low temperatures during the cycling.	No change in resistance. No evidence of abrasion of glass by expansion and contraction of the coils.
16.	Mycalex Corporation No. 555 glass-bonded mica	Samples exposed to Hg liquid and vapor for 100 hours at 600°F.	No evidence of penetration under 40 x magnification.
17.	Micalex Corporation No. 560 glass-bonded mica	Samples exposed to Hg liquid and vapor for 100 hrs at 600°F.	No evidence of penetration when observed under 40 x magnification.

11.0 CONCLUSIONS

The SNAP I nonmetallic materials development program made available materials and processing procedures that enabled uncooled high performance electrical machinery to operate reliably in a 550°F mercury vapor environment. The successful completion of a 2510 hour full power endurance run demonstrated the integrity of the alternator stator in this environment.

The sealing and insulating materials successfully used are not sensitive to nuclear radiation, although this was not proved by test.

Several materials are available for use as electrical insulation and potting compounds in the SNAP I environment. Some of the commercially available potting compounds have shown the required thermal and mercury compatibility though porous to mercury. The Mycalex glass-bonded mica compounds, Corning Pyroceram and mineral filled Dow Corning DC-7521 resin have demonstrated both thermal and mercury compatibility and the ability to act as barriers to mercury.

The primary problem area still existing is to determine processing techniques by which these materials may be consistently incorporated into a leak-tight stator assembly. In addition the program has pointed to several classes of materials useful for this type of application, and that the evaluation of these materials has only been touched upon. More work is required to fully exploit the pioneering provided by this program.

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The Technical Memoranda Reports (References 9 through 19) are not available for general distribution. Any inquiries concerning the availability of this information should be directed to the AEC.