MATERIALS EXAMINATION OF MODEL
SODIUM HEATED STEAM GENERATOR

SEP 13 1965

PROGRESS REPORT

MASTER

AEC CONTRACT AT(11-1)-1280

RESEARCH REPORT NO. 5131

BY: E. FORTNER
P. J. KOVACH

AUGUST 27, 1965

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MATERIALS EXAMINATION OF A MODEL SODIUM
HEATED STEAM GENERATOR - PROGRESS REPORT

By: E. Fortner
P. J. Kovach

ABSTRACT

PURPOSE

The purpose of this test program is to assess the compatibility of Croloy 2-1/4 and TP 316 SS in a liquid sodium system, particularly with reference to the design parameters of a full-size sodium-heated steam generator. This report contains the results of a metallurgical examination of piping removed from the model steam generator during intermediate shut-downs. Final interpretation of the data will be made after completion of a destructive examination of the model, to be described in a following report.

BRIEF OF TESTS

The samples were as-received commercial pipe and sections of pipe removed from the model steam generator after 1000, 2000 and 4000 hours of operation. The Croloy 2-1/4 was at 1000F. TP 316 SS pipe was removed from two positions, one at 1000F and the other at 1100F. Metallographic and microhardness studies were made of each sample. As-received and 4000 hour samples of Croloy 2-1/4 and 316 SS (1100F) were analyzed with the electron microprobe X-ray analyzer. Two special heat treatments were used with selected specimens to substantiate conclusions based on examination of the loop material.

RESULTS

The as-received Croloy 2-1/4 was slightly decarburized and contained surface depletions of chromium and silicon. Nickel and copper were piled up near the surfaces. These initial gradients (attributed to oxidation during mill treatment) and tempering effects complicate assessment of mass transfer processes.

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SODIUM HEATED STEAM GENERATOR

(Continued)

ABSTRACT (continued)

Slight decarburization (detectable to about 15-20 mils after 4000 hours), copper depletion, and nickel pick-up occurred during exposure.

No preferential mass transfer was indicated by the examination of the TP 316 SS samples although a general attack is probable. The material is characterized by sensitization during exposure and by persistent banding.

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INTRODUCTION

Sodium Heated Steam Generator

The Babcock and Wilcox Company is operating a model sodium-heated steam generator as part of the research support for the development, under AEC Contract AT (11-1)-1280, of a large sodium-heated steam generator of improved design. The model is constructed of the same materials proposed for the full-size unit, Croloy 2-1/4 and TP 316 SS. Insofar as possible, the surface to volume ratios, mass flow rates, temperature gradients across tube walls, and other physical and operating parameters of the model have been selected to simulate the full size steam generator. The details of the design, construction and operating history of the model steam generator have been given in B&W Research Reports by D. W. Koch.^{1,2}

Mass Transfer

The circulation of liquid metals at elevated temperature usually results in observable dissolution of the constraining walls at some positions and subsequent deposition of material at other positions. These phenomena may appear in many guises, all of which may be called mass transfer, if temperature gradients, composition (activity) inhomogeneities, or both are present. Dissolution may occur either as a rather uniform removal of metal or, more commonly, as a preferential leaching of certain constituents. Grain boundaries may be preferentially attacked. Deposition may occur in several ways, including physical plugging.

Mass transfer is fairly well understood, qualitatively. Diffusion rates, composition limits and activities within the various phases, possible interface controls or diffusion barriers, and especially the temperature dependence of the aforementioned are generally recognized as being of critical importance

in determining the type or extent of mass transfer. At present, however, both lack of basic information and the complexities of geometry, temperature distribution, the large number of chemical species involved, and so forth, make predictions of the extent of mass transfer in a realistic steam generator uncertain.

Transfer of carbon from the ferritic Croloy 2-1/4 to the austenitic 316 SS is of particular concern because this decarburization-carburization reaction is expected, on the basis of previous work,^{3,4} to be the fastest and most extensive process to occur in the secondary sodium system. Tube dimensions are not seriously changed by this process; carbon transfer resembles a leaching rather than uniform dissolution. However, the properties of both materials may deteriorate, the decarburized Croloy 2-1/4 becoming weaker and the stainless steel becoming embrittled. If significant fractions of wall thickness experience this type of transfer, serious loss of structural integrity may result. Mass transfer involving wall thinning can lead to premature failure because of increasing stresses associated with reduced cross sectional areas.

Purpose and Experimental Approach

Operation of the model sodium heated steam generator is a proof test for a proposed combination of materials and operating conditions. Following 8000 hours of operation, the model will be destructively examined to determine the extent of mass transfer.

Small sections of piping have been removed during intermediate shutdown of the model to check the progress of any mass transfer at two positions. This report presents the information derived from these samples. Final interpretation of the model test program and the assessment of mass transfer must await the more extensive results of the final destructive examination.

EXPERIMENTAL PROCEDURES

Sample History

The exposed samples discussed in this report are sections of seamless commercial 1-1/4" schedule 40 pipe removed from two isothermal U-bend sections of a model sodium heated steam generator during intermediate shutdowns following 1000, 2000 and 4000 operating hours. The locations of the high temperature (1100F) and low temperature (1000F) U-bends are indicated in the schematic plan, Figure 1. Figure 2 describes the arrangement of the model steam generator. The 1100F U-bend was constructed of a single section of 316 SS. The 1000F U-bend contained a welded juncture between 316 SS and Croloy 2-1/4. The Croloy 2-1/4 sample was at the maximum temperature for this material. Similarly the 1100F 316 SS samples were from the maximum temperature zone for this material. Samples of the as-received pipe used in these sections have also been examined.

During the scheduled shutdowns, the sodium was drained from the steam generator and replaced with an inert gas. The U-bends were removed and rinsed with alcohol. Sections of pipe, about 3" to 4" long, were sawed off each leg of the U-bends. The remaining portions of the U-bends were welded in place and reinsulated. The hot and cold traps have maintained the oxygen content of the sodium at less than 30 ppm, as indicated by plugging temperature.

Thus, four samples of piping were removed during each shutdown: two samples of 316 SS (which were essentially duplicates) from the 1100F U-bend and one sample each of 316 SS and Croloy 2-1/4 from the 1000F U-bend.

Two special 316 SS samples were prepared by heating pieces of the as-received pipe in air at 1250F for 16 and 48 hours. These special samples demonstrated the susceptibility of the 316 SS to sensitization, thus accounting

for observed carbide formation in the loop samples. Small samples of each of the four Croloy 2-1/4 conditions were given a special austenitizing heat treatment (1650F, 15 minutes, rapid cool) in a vacuum furnace. The special Croloy 2-1/4 samples were prepared to minimize the tempering effects found in the loop samples but to maintain any carbon gradients. Tabulated identification of the samples is given in Table I.

Specimen Preparation

The inner surfaces (the sodium side) of all pipe samples were visually examined. Longitudinal and transverse sections of each sample were separately wrapped in silver foil and mounted in Bakelite. The mounted specimens were polished using standard procedures for metallographic preparation.

Photomicroscopy

In general, standard etching and photomicroscopy practices were followed. Except as noted, Croloy 2-1/4 specimens were etched in Nital, the 316 SS specimens in Marble's reagent. Although both transverse and longitudinal sections were examined, in most cases only the microstructures of the transverse sections were recorded at both 100X and 500X. Additional photomicrographs were made when other magnifications, etches, or the recording of special features seemed appropriate.

Hardness Measurements

Diamond Pyramid Hardness measurements, using a 100 gram load and standard techniques, were made on the four Croloy 2-1/4 samples and the four 316 SS (1100F) samples. Two traverses were made on each specimen from the inner surface to about 40 mils from the surface. About 30 indents were made on each Croloy 2-1/4 specimen, about 15 on each of the 316 SS specimens.

Several hardness traverses were also made across the special austenitized Croloy 2-1/4 specimens, using a Knoop indenter with a 100 gram load.

All microhardness measurements were made on polished and lightly etched surfaces so that microstructural features could be observed during testing.

Microprobe Examination

The as-received and 4000 hour metallographic specimens of Croloy 2-1/4 and 316 SS (1100F) were repolished and examined by electron microprobe X-ray analysis. Point counting was employed in one micron steps from the inner surface to roughly one mil from the surface. Several traces were made on each specimen, three elements being analyzed with each trace. Several continuous profile scans were made over much larger distances, in many cases extending from the inner surface about 25 mils. Analyses were made on both materials for iron, chromium, nickel, silicon, molybdenum and copper. Backscattered electron traces were made with most point counting runs to pinpoint the surface position. Copper and nickel analyses of the Croloy 2-1/4 were also made near the outer surface. Copper and nickel contents of the Croloy 2-1/4 were determined from an appropriate series of standards. Microprobe readings at the interior of the tube were normalized to the bulk chemical analysis to determine percentages of other elements.

OBSERVATIONS AND RESULTS

Croloy 2-1/4

a) Summary

A slight gradual decarburization of Croloy 2-1/4 exposed to 1000F sodium in this system has been detected. Tempering effects and original gradients

complicate exact assessment of the rather small carbon loss detected. Partial decarburization is metallographically detectable to about 10 mils after 4000 hours. The greater affected depth indicated by micro-hardness measurements involves a very small carbon loss and may be considered of little significance.

Microprobe results indicate pick-up of nickel (to about 1% at the surface) and copper depletion during exposure. Leveling out of initial chromium and silicon gradients was also observed, part of which may be actual pick-up.

b) Macroscopic

Visual examination of the inner surfaces (sodium side) of the removed pipe samples did not reveal any signs of mass transfer. Neither deposits nor dendritic growths were apparent. Figure 3a illustrates the appearance of the sodium side of Croloy 2-1/4 after 4000 hours at 1000F.

c) Microstructure

The microstructure of the ID surface region of the as-received Croloy 2-1/4 piping is shown in Figure 4a. This can be compared with Figures 4b, 4c and 4d, which show the microstructure after 1000, 2000 and 4000 hours at 1000F respectively.

The carbides in the as-received mill processed piping are, for the most part, in colonies of poorly defined pearlite. The apparent area per cent carbides on the polished and etched surface slightly decreases between the interior and inner surface of the pipe, indicating a slight decarburization during manufacture of the pipe.

The most obvious structural changes during loop operation are attributed to the tempering effects of exposure at 1000F for long periods of time.

Although these changes are visible in Figures 4a through 4d, Figures 5a and 5b of the center of the as-received and 4000 hour specimens illustrate the tempering effects more clearly. The carbide colonies have quite different appearances as carbides gradually spheroidize and presumably change composition as well as size and number with increasing time at temperature. The development of a dot-like carbide in what was initially clear ferrite grains is noteworthy. Tempering changes are unrelated to the environment and must not be attributed to the sodium in the system. However, tempering may influence the course of any decarburization and certainly complicates metallographic assessment of decarburization.

No completely carbide free rim has developed even after 4000 hours exposure to sodium at 1000F. The apparent progressive decrease in area per cent of carbides on the metallographically prepared surface between unaffected interior and surface, as illustrated in Figure 4, does indicate partial decarburization. The actual carbon loss to account for these changes, which are detectable to about 10 mils after 4000 hours (with questionable detection, in view of the complications, to perhaps 15 mils) and a lesser distance after 2000 hours, is small. Detection of carbon loss in the 1000 hour specimen is uncertain.

The microstructures of the briefly austenitized specimens consisted of ferrite in the surface regions and bainite in the interior, the transition region consisting of a mixed structure. The small ferrite rim on the as-received material indicates that this material was initially decarburized. The exposed samples had, as a group, a larger ferrite rim although an increased depth with increasing exposure time is detectable. The microstructures of two of these special samples are shown in Figure 6.

d) Microhardness

The results of microhardness testing of the Croloy 2-1/4 series of loop specimens are summarized in Figure 7. The bar graphs were derived from envelopes of the original data, plotted as hardness versus distance. The large scatter for each specimen is for the most part real. The hardness depends on the structure, which may be viewed as a non-uniform dispersion of hard carbides in a soft ferrite matrix, in the immediate vicinity of the indent. At almost any distance from the surface an indent might fortuitously be made in a carbide free region or conversely might land on a local carbide colony. However, the probability of encountering carbides decreases as one approaches the decarburized surface.

The extrapolated surface hardness of all exposed specimens is about the same (145 DPH). This may be taken as the characteristic ferrite hardness. The tempering effects are shown by the decreasing hardness with increasing exposure time of the interior of the pipe which is not influenced by the presence of sodium. The decrease in width of the scatter band is another indication of tempering. The gradual drop in hardness between interior and surface of the specimens is an indication of the extent of decarburization although the affected depth is difficult to exactly specify because of the scattered nature of the data and because no natural demarkation exists between unaffected material and decarburized material.

The special austenitized specimens were prepared to minimize the hardness scatter resulting from the type of carbide distribution in the loop samples and to minimize the complicating tempering effects which are of only passing interest. Results of microhardness traverses across these special specimens are summarized in Figure 8. As in Figure 7, the results are shown in bar

graph form, the envelopes of the original data being used to construct the bars. Some leveling out of carbon gradients may be expected during austenitizing. However, the data show that carbon redistribution has been local; that is, the carbide-ferrite aggregates of the untreated samples have been for the most part replaced by a more uniform structure, bainite, but gradients occurring over several mils are still detectable. The hardness decrease near the surface of all specimens is a measure of decarburization. The area between curves on a hardness-distance plot is a better measure of carbon loss than a distance of affected depth. Thus, although an almost constant hardness, for all samples, does not occur until some 25 mils from the surface, clearly a progressive decrease, with time, of the area under the hardness-distance curves does indicate slight gradual carbon loss. It should be noted that only differences between exposed and as-received material may be attributed to carbon loss during exposure. A significant part of the gradients must be attributed to the original decarburized condition. Hardness differences at depths greater than 20 mils are of little consequence, considering residual tempering differences and probable experimental data scatter. Small hardness gradients well away from the surface may be attributed to the small temperature gradient across the pipe wall during operation. This would cause a "tempering gradient" which would result in a slight hardness decrease between inner and outer pipe surfaces, independent of other effects.

e) Microprobe

Microprobe results of a point count examination of the sodium side of the as-received and 4000 hour samples are summarized in Figures 9a and 9b respectively. The many scanning traces gave no more real information than is contained in Figure 9; that is, composition throughout most of the material

is quite uniform except within about 0.3 mils of the surface. Thus, the more accurate point counting results are given greater emphasis. The gradients in the surface region of the original material are attributed to diffusion which occurred during oxidation during heat treatment in the manufacture of the pipe. The chromium and silicon would be expected to be preferentially oxidized depleting, as observed, the material next to the oxide. Nickel and copper would be rejected from the oxide, preferring to remain in the metallic constituent, and thus are piled up next to what was at one time an oxide-steel interface. Because of the vagaries of oxidation, exact reproduction of the data obtained on rather widely separated traces should not be expected. However, quite similar gradients were found near the outer surface of the as-received material.

When examining the composition gradients in the 4000 hour specimen one must, as with the decarburization information, compare the final gradients with the pre-existing gradients. Thus, although the surface of the 4000 hour sample is depleted in chromium, substantial chromium enrichment (compared with the original surface) is nonetheless indicated. A surface peak would normally be expected if chromium were being picked up by the steel. The absence of such a peak may be indicative of a small availability of chromium in the sodium, indicating a rather low chromium activity. However, the contribution of a simple diffusion leveling out of the chromium gradient is uncertain and may account for the bulk of the observed changes. Similar comments apply to the leveling out of the silicon gradient. The nickel peak and copper depletion are indications of mass transfer. However, the effects are certainly small, although in the direction anticipated.

No indication of composition changes of a magnitude needed to cause austenite formation has been found. As in the as-received material, only slight variations from trace to trace were found, and this must include any effects of the inherited differences in initial gradients. It is appropriate to consider these differences between traces over different regions as real and thus to study individual traces rather than the average of several separated traces. The data presented were obtained from repeated traces over the same area; incomplete coverage of other regions was made to check the typical aspect of the information. The changes may be represented by comparing surface compositions (at a depth of 1 micron) with the average composition (at depths greater than 9 microns) as given in Table II.

316 Stainless Steel

a) Summary

No preferential mass transfer effects have been found in the 316 SS pipe, although some uniform removal of material is probable. The steel does sensitize readily and is further characterized by banding, both phenomena being unrelated to the sodium environment.

b) Microstructure

The microstructures of the as-received and 4000 hour specimens exposed at 1100F are illustrated in Figure 10 (100X) and Figure 11 (500X). The different etches used on exposed and as-received material should be noted. The as-received material was difficult to etch, grain boundaries being outlined only after a relatively long etching time. On the other hand,

the grain boundaries of all exposed specimens were easily delineated, although apparent width generally increased with increasing time.

Note the banding effect persistently observed in all specimens and the change in surface profile shown in Figures 10a and 10b. The evidence of extensive deformation of the surface region in the as-received material, illustrated in Figure 11a, is especially noteworthy. The gradual precipitation of carbides during exposure is attributed to sensitization, not to carburization. This familiar process is the precipitation of carbon, as carbides, from the originally supersaturated austenite, preferentially at grain boundaries and twin boundaries but also, in advanced stages, within the grains in a Widmanstätten pattern. Figure 11b shows the typical appearance of the 316 surface regions after 4000 hours at 1100F when polished and lightly etched. Note that the thicker appearing grain boundaries occur to a depth similar to the depth of heavy distortion in the as-received material. Figure 11c, with a slightly heavier etch, shows a less typical but not rare area in which the Widmanstätten effect is quite apparent. To substantiate the attributing of carbide formation to carbon present in the as-received material, specimens of as-received material were heated in air at 1250F for 16 hours and 48 hours respectively. The structures of these two special specimens are shown in Figures 12a and 12b. Allowing for the effect of differences of temperature, the same microstructural features formed during exposure in the model steam generator are developed. Thus, carburization is not indicated, although the very first indication of carbon pick-up might be masked by the more obvious sensitization. That sensitization occurs throughout the pipe wall demonstrates that this process can not be attributed merely to carburization.

Especially noteworthy is the advanced sensitization near the surface of the special specimens as well as the loop specimens. This is attributed to the known accelerating effects of the deformation observable near the inner surface of as-received material.

The 1000 and 2000 hour specimens and all specimens exposed at 1000F look rather similar. The grain boundaries etch quite readily. All specimens appear to be undergoing sensitization, the process being more advanced in specimens exposed for longer times or at the higher temperature.

No evidence of preferential mass transfer is metallographically detectable. However, changes in the surface profile during exposure indicate that some uniform attack has probably occurred. This may be a non-recurring attack of the initial pickled surface. Once susceptible grains are removed, the attack stops. Figure 3b illustrates the macroscopic appearance of the sodium side of the 316 SS pipe after 4000 hours at 1100F.

c) Microhardness

Microhardness traverses of the 316 SS (1100F) specimens are summarized in Figure 13. No data points were more than 5 microhardness units away from the curves. The initial high surface hardness substantiates the metallographic evidence of a cold worked surface. The gradual decrease in both surface hardness and interior hardness may be called a tempering effect. Carbon, originally dissolved in the austenite, contributes to grain hardness. Removal of carbon as rather ineffectual grain boundary carbides softens the steel. Recovery in strained regions also contributes to the softening. Removal of the initially harder surface grains by mass transfer could contribute to the lower hardness of the exposed piping at the new surface.

d) Microprobe

No mass transfer is indicated by the microprobe examination of the 316 specimens. Figure 14 summarizes the results for the 4000 hour specimen obtained from repeated traces over the same path. The banding or segregation of nickel, chromium, and to a lesser extent molybdenum is apparent. Averages of several traces would obviously obliterate the banding details, yielding only average percentages. The maximum and minimum chromium and nickel contents were plus and minus about 1/2% of the average concentration. Iron, of course, also shows segregation to make the total metal content remain constant. As observed metallographically, the banding occurs throughout the steel. However, small regions of a mil or so are apparent which do not have this characteristic.

No probe traces indicated mass transfer effects, although naturally, because of the nature of the banding, different probe traces yielded slightly different surface compositions. Because of the gradients (from banding) extending to the surface the very initial stages of preferential mass transfer (say within 0.1 mil) might be masked.

The total metallic content of the as-received surface dropped off slightly in a region within some 0.1 mils from the surface, due to the appearance of small gradients in all constituents. A tentative explanation is that a sub scale, formed during mill treatment and remaining after pickling, is present. This effect is not detectable in the exposed specimen, substantiating the probability of a uniform removal of surface material.

DISCUSSION

I. Croloy 2-1/4

Detection and assessment of the small observed changes in carbon content, attributable to mass transfer, is complicated by the obvious tempering effects and the initial composition gradients. After 4000 hours, the affected depth is metallographically discernable to about 10 mils. The major hardness gradients also occur within this distance. Although distinct hardness differences are present to roughly 20 mils, slight differences at greater depths may be of little significance. Although the U-bends are described as "isothermal", a small radial temperature gradient may have resulted in lesser tempering with increased distance from the sodium side, thus contributing slightly to the apparent continued increase of microhardness with depth. This partial decarburization, which represents only a small carbon loss, is perhaps better described by a measure such as the area between curves on a hardness distance plot if, as for the special specimens, the hardness at the unaffected interior is similar for all specimens, thus minimizing the tempering effects. Using this measure, only a fraction of the total carbon within the affected region has been removed, as also shown metallographically.

Because of the scatter of the microhardness data expected in this type of microstructure, designation of a depth of decarburization when no natural demarkation exists between unaffected and affected material is tenuous. The changes in visual appearance of the microstructure due to tempering induced changes in actual carbide characteristics and etching behavior must be remembered when visually assessing the area per cent carbides, which is a measure of carbon content. Similarly, the usual analysis techniques for

for determining the percentages of various constituents are of little assistance.

The loss of copper from Croloy 2-1/4 has not been, to our knowledge, previously reported although the small amounts of this trace element are not apt to be of engineering significance. Nickel pick-up is also a mass transfer process. The increase in chromium and silicon contents after 4000 hours must be interpreted with caution because the contribution of natural leveling out of the original gradients is difficult to determine. In any case, the amounts of transferred material are not nearly enough to cause austenite formation and the affected depth is small. Although mass transfer of these heavier elements is not apt to change bulk properties of piping, such transfer may influence the course of the more rapid carbon transfer and thus indirectly exert an influence of engineering concern. Pick-up of the heavy elements is apparently controlled by the behavior of the source, 316 SS. The absence of detectable preferential depletion in the 316 SS pipe examined correlates with the small pick-up by Croloy 2-1/4 of nickel and chromium. However, a more extensive 316-sodium interaction, caused by a rise in temperature for instance, might very well change the behavior of Croloy 2-1/4 at 1000F. The system must be viewed as a whole; events at one portion of the loop can control behavior at another portion.

II. 316 SS

The interesting banding and sensitization effects are only incidental to the assessment of mass transfer in the model steam generator. The differences between commercial pipe and carefully prepared laboratory samples may influence observed mass transfer effects. Susceptability of pickled

surfaces to attack by sodium, influence of a possible subscale, and the effects of a deformed surface are not commonly studied with prepared samples and are indeed generally considered of only slight importance. However, the importance of surface condition is recognized as being of critical importance in other "mass transfer" processes such as nitriding and carburizing.

The absence of preferential mass transfer is significant because material examined was at the highest temperature in the system. Possible down-stream effects and the indication of some uniform attack need to be further evaluated.

III. General

The examination of pipe samples has been made to monitor the mass transfer effects throughout the system. Results are encouraging from the view of confirming the compatibility of materials and over-all operating conditions, although larger changes would be easier to describe and numerically assess with confidence. The final destructive examination should provide more definitive information, not only because of the more extensive changes probable after longer times, but also because the entire system can be examined with an aim of evaluating possible down-stream effects and perhaps even attempting a form of mass balance, although the latter would be a quite lengthy and tenuous procedure.

The results must not be viewed simply as showing the behavior of Croloy 2-1/4 in 1000F sodium or the behavior of 316 SS in 1100F sodium. The type of mass transfer at a local region depends on the nature of the sodium in contact with the steel as well as the characteristics of the steel. Although

processes within the steel, such as diffusion with the associated strong dependence on temperature, may well be local rate determining steps; the direction of preferential transfer and driving forces depend on the activity of the various constituents in the sodium as well as in the steel. It is this latter factor which accounts for the necessity of viewing the system as a whole because of the interaction of processes occurring at widely separated positions.

A continuing mass transfer obviously requires a source and a sink. Preferential transfer of carbon, for instance, requires many steps. Carbide disintegration to feed carbon to a matrix, diffusion to the sodium-steel interface, transfer across the interface, transfer through the sodium and essentially the reverse processes of carbon pick-up must all occur. Over-all rates are commonly identified with a slowest step. However, the relative surface and volumes of source and sink also are of critical importance; a small ferritic sample in an austenitic sodium loop will behave quite differently from the same ferritic material in the form of a similar loop, containing a small section of the austenitic material. Failure to identify copper and carbon sinks in the examined sections is not of great concern. The small amounts of material removed may either be residing in the sodium or may be discovered at other locations during the more complete destructive examination.

These comments are indications of some of the considerations to be made in interpreting the results presented and as a general rationale of the approach to the assessment of a particular system.

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Submitted by: E. Fortner
E. Fortner

P. J. Kovach
P. J. Kovach

Approved by: J. P. Rowe
J. P. Rowe

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TABLE I

Sample Identification

Original Composition*

	<u>C</u>	<u>Cr</u>	<u>Ni</u>	<u>Mo</u>	<u>Si</u>	<u>Cu</u>	<u>P</u>	<u>C</u>	<u>Mn</u>
AISI 316 SS	0.05	17.30	13.72	2.30	0.47	0.14	0.019	0.007	1.67
Croloy 2-1/4	0.11	2.30	0.12	1.00	0.35	0.09	0.013	0.015	0.45

Thermal History			Special Examination	
	Temp (°F)	Time (hours)	Microhardness	Microprobe
Croloy 2-1/4				
1	--	0	✓	✓
2	1000	1000	✓	
3	1000	2000	✓	
4	1000	4000	✓	✓
5	--	0**	✓	
6	1000	1000**	✓	
7	1000	2000**	✓	
8	1000	4000**	✓	
316				
9	--	0	✓	✓
10	1000	1000		
11	1000	2000		
12	1000	4000		
13	1100	1000	✓	
14	1100	2000	✓	
15	1100	4000	✓	✓
16	1250	16		
17	1250	48		

* Mill analyses (except Cu and Ni in Croloy 2-1/4, which were microprobe analyses).

** Plus 1650F, 15 min, rapid cool.

TABLE II

Outline of Microprobe Results for Croloy 2-1/4

	<u>1 Micron from Original Surface</u>	<u>1 Micron from Exposed Surface (4000 hours, 1000F)</u>	<u>Interior</u>
Wt % Cr	0.08	1.98	2.30
Mo	1.00	1.00	1.00
Si	0.10	0.24	0.35
Ni	0.22	1.00	0.12
Cu	0.39	Not Detectable	0.09

SCHEMATIC OF MODEL SODIUM-HEATED STEAM GENERATOR

ARRANGEMENT OF SODIUM HEATED MODEL STEAM GENERATOR

Figure 3 - Sodium Side of Pipe After 4000 Hours
Exposure to Liquid Sodium 3.2X

a) Croloy 2-1/4, exposed at 1000F

Figure 3 - (Continued)

b) 316 SS, exposed at 1100F

a) 0 hours (as-received)

b) 1000 hours

Figure 4 - Microstructure of Surface Region of Croloy 2-1/4 Pipe
Exposed to 1000F Sodium. 250X Nital Etch

c) 2000 hours

d) 4000 hours

Figure 4 - Continued

a) 0 hours (as-received)

b) 4000 hours

Figure 5 - Microstructure of Interior of Croloy 2-1/4 Pipe to Show Tempering Effects of Exposure at 1000F Unrelated to the Sodium Environment. 500X Nital Etch

Nital 100X

As-Received and Heat Treated for 15 mins at 1650F

Nital 100X

Exposed in loop for 4000 hours at 1000F and
Subsequently Heat Treated for 15 mins at 1650F

Figure 6

MICROHARDNESS OF CROLOY 2 $\frac{1}{4}$. DIAMOND PYRAMID HARDNESS (100 GRAM LOAD) NEAR THE INNER SURFACE OF PIPE EXPOSED TO 1000F SODIUM

MICROHARDNESS OF SPECIAL CROLOY 2 $\frac{1}{4}$. KNOOP HARDNESS (100 GRAM LOAD) NEAR THE INNER SURFACE OF PIPE EXPOSED TO 1000F SODIUM AND SUBSEQUENTLY AUSTENITIZED

MICROPROBE ANALYSIS OF AS-RECEIVED CROLOY 2 $\frac{1}{4}$

FIGURE 9 a

MICROPROBE ANALYSIS OF CROLOY 2 $\frac{1}{4}$ EXPOSED TO 1000F SODIUM FOR 4000 HOURS

FIGURE 9 b

a) 0 hours (as-received)

b) 4000 hours

Figure 10 - Microstructure of 316 SS Pipe to Show Change in Surface Profile During Exposure to 1100F Sodium and Persistent Banding. 100X a) 70% HNO_3 , Electrolytic; b) Marble's Reagent.

a) 0 hours (as-received) Note Deformation Markings

Figure 11 - Microstructure of Surface Region of 316 Pipe
Exposed to 1100F Sodium. 500X

a) 70% HNO_3 , Electrolytic;
b) and c) Marble's Reagent

b) 4000 hours Typical Sensitization.

c) 4000 hours Advanced Surface Sensitization.

a) 16 hours

b) 48 hours

Figure 12 - Microstructure of 316 Pipe After Special Sensitizing Treatment in Air at 1250F. 500X Marble's Reagent

MICROHARDNESS OF 316 SS. DIAMOND PYRAMID
HARDNESS (100 GRAM LOAD) NEAR THE INNER
SURFACE OF PIPE EXPOSED TO 1100F SODIUM

FIGURE 14

MICROPROBE ANALYSIS OF 316 SS EXPOSED TO 1100F SODIUM FOR 4000 HOURS