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DATE: June 2, 1955

SUBJECT: EXAMINATION OF INCONEL-316 STAINLESS STEEL-SODIUM PUMP
LOOPS 4689-5 and 4689-6

CLASSIFICATION CANCELLED
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EXAMINATION OF INCONEL-316 STAINLESS STEEL-SODIUM PUMP LOOPS 4689-5 and 4689-6

G. M. Adamson, R. S. Crouse

These are two more of the series of loops operated by Experimental Engineering at the request of Metallurgy. These two loops were constructed with differing amounts of 316 stainless steel in the cold leg. In loop 4689-6, both the economizer and the cold loop were of 316, while in 4689-5 only the cold loop was 316. An all Inconel control loop for this series was 4689-4 which has been previously reported, CF 55-4-167.

The engineering variables on these loops as furnished by D. R. Ward, the designer and operating engineer from Experimental Engineering, are listed in Table I. The location of the metallographic samples, the configuration and temperature pattern of loop 4689-5 are shown in Figure 1 with similar data for 4689-6 in Figure 2. Both loops had been cleaned by a prior circulation of sodium. Both loops were also dried by heating under vacuum before any filling was done. Loop 4689-6 was cleaned by approximately 16 hours of isothermal operation with temperatures between 850 and 900°F followed by 2 1/2 hours at 1300°F with the sodium being dumped while still hot. During the cleaning fill for 4689-5, the sodium was accidentally contaminated by air and plugged the loop before filling was complete. The loop was then drained and cleaned with butyl alcohol and rinsed with acetone. After being dried and filled with helium, it was then refilled with sodium and the same cleaning cycle used in -6 was followed.

Exam. of Inc.-316 S.S.-Sodium Pump
Loops 4689-5 and 4689-6

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TABLE I

Engineering Variables on Pump Loops 4689-5 and 4689-6

Loop No.	4689-5	4689-6
Operating Time - Hours	1000	1000
Maximum Sodium Temperature, °F	1500	1500
Maximum Wall Temperature, °F	-	-
Sodium ΔT , °F	300	300
Reynolds Number - Hot Leg	66,000	66,000
" " - Cold Leg	58,000	58,000
" " - Economizer Annulus	310,000	310,000
" " - Economizer Center	83,000	83,000
Velocity, Hot and Cold Legs, ft/sec	3.0	3.0
" , Economizer Annulus, ft/sec	8.4	8.4
" , Economizer Center, ft/sec	5.5	5.5
Length of Heater - Inches	110	110
" " Cooler - Inches	165	165
" " Economizer - Inches	110	110

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Discussion

Both of these loops showed hot leg attacks similar in nature to that which has been reported for 4689-4. In loop -5 it was as heavy intergranular penetrations to 2 1/2 mils, Figure 3. A similar attack to 1 1/2 mils was found in -6, Figure 4.

Heavy layers were found in the economizers of both of these loops. Photographs of sections from identical areas in the economizer and cold legs of these loops and also from 4689-4 are offered for comparative purposes in Figures 5 and 6. In these macro photographs the upper three sections are from the inner tube of the economizer, in order of decreasing temperature, while the bottom section is from the cold leg. In loop -5 the maximum layer was to 15 mils and was in the cold end of the center tube of the economizer. Both by spectrographic means and by metallographic etching it was shown that the crystals are predominantly nickel. In the cold leg of this loop the deposit was much thinner than was found in the economizer. A diffusion zone primarily in the grain boundaries and up to 5 mils thick, as well as the layer, was found on the surface of the 316 sections, Figure 7. Figure 8 is a photomicrograph showing this diffusion and resulting alterations in the grain boundaries in the 316 economizer.

The deposit in the economizer of loop 4689-6 was similar to those in the other loops and had a maximum thickness of 12 mils. No deposit was found metallographically in the last half of the cold coil; however, scattered crystals were found visually. A diffusion layer was present wherever the layer was found on a 316 surface. At the 316 stainless steel-Inconel

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junction at the hot end of the economizer, the Inconel was attacked to 3 mils while the 316 was unattacked.

The chemical results available on these loops are tabulated below. No samples were taken as the sodium was charged but analysis from the sodium in original drum are: O₂ - 300 ppm; Ni - 3 ppm; Fe - 10 ppm; Cr - 9 ppm. After operation, one sodium sample was taken in a hydrogen cleaned metal sampler as the loop was drained and a second was taken in a glass bulb as soon as the sump had cooled sufficiently. The results of these samples are tabulated below:

Loop No.	Type of Sampler	Oxygen ppm	Nickel ppm	Chromium ppm	Iron ppm
4689-5	Metal	300	30	35	35
-5	Glass	280	5	4	10
-6	Metal	290	15	7	10
-6	Glass	290	-	-	-

These analyses would say that there is essentially no change in either oxide or impurity content during the run; however, from the reproducibility figures now being obtained on oxide analyses in sodium, it is obvious that these figures must be taken with a grain of salt. These loops both operated with diffusion type cold traps which should remove most of the oxides in 1000 hours of operation. The low values of nickel, chromium and iron are what would be expected from available solubility data.

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Exam. of Inc.-316 S.S.-Sodium Pump
Loops 4689-5 and 4689-6

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Conclusions

These two loops confirm the fact noted with 4689-4 that excessive mass transfer is occurring in Inconel pump loops in which sodium is circulated at 1500°F.

When these loops were scheduled, the large amounts of mass transfer found were not expected. A close examination of the cold leg does show a decreasing amount of deposit with increasing 316 cold surface; however, because of the excessive amount of mass transfer, this may be due to variations in other variables. At least, it appears that the use of 316 stainless steel in the cold leg of the loops does not increase mass transfer.

Because of doubt in the analyses, no conclusion may be drawn on the role of the oxides.

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GMA:h

Attachment

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APPENDIX

METALLOGRAPHIC EXAMINATION OF SODIUM PUMP LOOP 4689-5

Loop 4689-5 - Inconel pump loop with 316 SS cold section - Run 1000 hours at 1500°F with Sodium. $\Delta T = 300^\circ\text{F}$.

X-7396 (1)

The surface was smooth with moderate intergranular penetrations to 1 1/2 mils.

X-7397 (2)

The surface was smooth with moderate to heavy intergranular penetrations to 2 1/2 mils.

X-7400 (3-0)

The surface was a bit rough with moderate intergranular penetrations to 2 mils. A second metallic phase was present in many of these penetrations.

X-7401 (3-I)

The inside surface of this section was rough with many small pits to about 1/2 mil deep. The outside was about like section (X-7400) above.

X-7638 (5a-I)

Sample lost.

X-7639 (6a-I)

Heavy metallic deposit to 15 mils thick on inside.

X-7398 (4-0)

The surface was a bit uneven but no deposit was seen.

X-7399 (4-I)

There was a heavy deposit of needle-like crystals in masses to 10 mils thick on the inside of the tube. These crystals were indistinguishable from the nickel plate until etched for nickel. No particular evidence of a deposit was seen on the outside of the tube.

X-7640 (7a-I)

Heavy metallic deposit to 8 mils on inside.

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X-7402 (-5)

A continuous metal deposit was present on the surface to 1/2 mil thick. The deposit was partially diffused into the stainless steel. Also, diffusion of some material into the stainless steel to a depth of about 2-1/2 mil had taken place. Whatever this material is, it has lowered the resistance of the stainless steel to aqua regia etching reagent to a marked degree, especially along the grain boundaries.

X-7403 (-6)

This section was very much like the one above except the deposit was somewhat thinner and the effect of the diffusion was about twice as deep.

X-7404 (-7)

This section also showed the effects of a diffused material to about 2 mils and an intermittent metal deposit on the surface much like the two above samples.

X-7405 (-8)

Moderate intergranular penetration to 1-1/2 mil deep.

X-7406 (-9)

Same as above.

X-7407 (-10)

Same as above.

X-7408 (-11)

On the inside surface of the outside pipe there was an occasional clump of metal crystals. The inside surface of the inner tube there was a very heavy deposit of needle-like crystals with masses to 10 mils thick.

X-7409 (-12-0)

Surface uneven but no other evidence of attack.

X-7411 (-12-I)

A deposit of needle-like crystals were present to a thickness of 11 mils.

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X-7410 (-13)

Some evidence of diffusion and an intermittent surface deposit.

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METALLOGRAPHIC EXAMINATION OF SODIUM PUMP LOOP 4689-6

Loop 4689-6 - 1000 hours at 1500°F with Sodium. Loop had an Inconel hot section and a 316 SS economizer and cold section. $\Delta T = 300^{\circ}\text{F}$.

T-7464 (-1)

Moderate to heavy intergranular attack to 1 1/2 mil deep.

X-7465 (-2)

Same as above.

X-7466 (-3)

Same as above.

X-7467 (-4)

Same as above.

X-7468 (-0)

No attack or deposit.

X-7469 (5-I)

No attack or deposit was seen on the inside of the tube and no deposit on the outside. The outside surface was a bit rough, however.

X-7642 (5a-I)

Scattered metallic crystals on inner surface.

X-7470 (6-0)

Surface a bit rough with some penetrations to 1 mil.

X-7471 (6-I)

Both the inside and outside of the tube are slightly rough and uneven but no deposit is visible.

X-7643 (6a-I)

Heavy layer of metallic crystals to 7 mils on inner surface.

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X-7472 (7-0)

Surface somewhat rough.

X-7473 (7-I)

A crystal deposit to 12 mils thick was present on the inside of the tube. These crystals were rather angular.

X-7644 (7a-I)

Heavy layer of metallic crystals to 8 mils on inner surface.

X-7474 (8-0)

Surface slightly rough. No deposit.

X-7475 (8-I)

A crystal deposit to 9 mils was present on the inside of the tube.

X-7476 (-9)

The surface was slightly rough with a few clumps of crystal present.

X-7477 (-10)

No attack or deposit.

X-7478 (-11)

No attack or deposit.

X-7479 (-12)

No attack or deposit.

X-7480 (-13)

The Inconel part of this junction was heavily attacked with a rough surface and intergranular penetration to 3 mils. The 316 SS was unattacked.

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Figure 1
Pump Loop 4689-5
Location of Metallographic
Samples and Wall Temperatures.

T-7850

12

T-7826 250X
Figure 3. Maximum Attack in Hot Leg of Inconel-316 Stainless Steel Pump Loop 4689-5 After Circulating Sodium for 1000 Hours.

T-7768 250X
Figure 4. Maximum Attack in Hot Leg of Inconel-316 Stainless Steel Pump Loop 4689-6 After Circulating Sodium for 1000 Hours.

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LOOP 4689-4

T-7761

Figure 5. Deposit in Cold Leg and Economizer of All Inconel Loop 4689-4 After Circulating Sodium for 1000 Hours.

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Inner Economizer
Tube

Cold
Leg

LOOP 4689-5

LOOP 4689-6

T-7759

T-7760

Figure 6. Deposits in Cold Legs and Economizers of Inconel-316 Stainless Steel Loops 4689-5 and 4689-6, After Circulating Sodium for 1000 Hours.

-10-

427-16

Protective
Plating

Layer

Diffusion
Zone

T-7716

250X

Figure 7. Layer and Diffusion Zone in Cold Leg of Inconel-316 Stainless Steel Loop 4689-5 After Circulating Sodium for 1000 Hours.

Protective
Plating

Deposited
Crystals

Surface
Layer

T-7713

Figure 8. Deposit in Inner Tube of Economizer From Pump Loop 4689-5 After Circulating Sodium for 1000 Hours.