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EXAMINATION OF COMPATIBILITY OF CAVITATION-RESISTANT MODIFICATIONS TO TYPE 316LN STAINLESS STEEL IN A MERCURY THERMAL CONVECTION LOOP

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S. J. Pawel, E. T. Manneschmidt

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S. J. Pawel
E. T. Manneschmidt

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ABSTRACT

A 316L stainless steel thermal convection loop (TCL) containing a variety of stainless steel coupons circulated mercury for 2000 h. The TCL conditions included a maximum temperature of 307°C, a maximum temperature gradient of 90°C, and a Hg velocity of about 1.4 m/min. In addition to mill-annealed/surface-ground 316LN coupons serving as the baseline material, other coupons included 316LN that was 50% cold-worked, 316LN that was given a proprietary surface hardening treatment termed "kolsterizing," and Nitronic 60. The purpose of this test was to examine Hg compatibility with these modest variations of annealed 316LN stainless steel that are considered potential improvements over annealed 316LN for cavitation-erosion resistance in the Spallation Neutron Source (SNS) target containment system. The results indicated negligible weight change for each coupon type, no significant indication of attack or surface roughening, and generally no interaction with Hg.

1. INTRODUCTION

The Spallation Neutron Source (SNS) will generate neutrons via interaction of a pulsed 1.0 GeV proton beam with a liquid mercury target. Type 316L/316LN austenitic stainless steel has been selected as the primary target containment material based on a favorable combination of factors, including resistance to corrosion and embrittlement by Hg, well-characterized behavior in a radiation environment, and the absence of a significant ductile-brittle transition temperature such as that found in irradiated ferritic stainless steels.[1]

In the SNS target, the incident proton beam will be pulsed. The duration of each pulse will be short (<1 ms) and the temperature rise of the affected volume will be small (a few °C), but the rate of temperature rise during each pulse will be exceptionally high (on the order of 10^7 °C/s). The energy deposition from each pulse will result in the expansion of the volume in which it occurs, and this is expected to give rise to a thermal-shock induced pressure wave, which then travels into the surrounding Hg. When the compression wave reaches a boundary (e.g., the container wall), it will be reflected back with a change of phase. The resulting rarefaction wave travels back into/through the Hg, exposing the Hg to transient negative pressures. At a sufficient negative pressure, microscopic bubbles are expected to form in the Hg. Previous research[2, 3] indicated less than one MPa is required to generate bubbles in Hg of nominal purity at SNS. When the bubbles collapse (in principle, with each pulse cycle) at/near the containment surface, some of the energy released – typically a "jetting" action of liquid at extreme velocity – can effectively erode the surface through a scrubbing action. Calculations [4] for SNS operating conditions suggest that negative pressures sufficient to induce cavitation will be routinely present in the target near the beam window, and therefore cavitation-erosion potentially could be a localized wastage issue for the SNS target container.

Some recent experiments [5-9] have indicated that pressure pulses in Hg appear capable of causing pitting and/or cavitation-erosion damage in stainless steel containers. For example, experiments in which stainless steel surfaces in contact with Hg were subjected to mechanically-induced pressure pulses via the Split-Hopkinson Pressure Bar (SHPB) technique generated shallow pits on the container walls. Subsequently, cylindrical Hg-filled containers with flat ends were irradiated with 200 pulses of 800 MeV protons at SNS-relevant beam intensities. In-beam experiments are still ongoing,[5] but the presence of pits on the flange ends of the containers was confirmed for several combinations of materials and surface treatments. The individual pits/clusters had various diameters and were generally on the order of 20 μm deep. Even though no

relation between the number of pulses and pitting damage has been established, cavitation erosion damage appears to be a potential issue for the mercury target containment given the design life expectation of perhaps 6-7 orders of magnitude more pulses for the SNS target than experienced by either the SHPB tests or in-beam exposures.

Alternatives to annealed 316L/316LN are being considered for the target container in an effort to increase resistance to cavitation-erosion. However, an important criterion for any alternative is that it be of a sufficiently similar alloy class to 316L/316LN that the accumulated data for 316LN concerning compatibility with Hg, fatigue properties, and radiation resistance would be expected to generally apply for the new material. Failing that, such data would have to be collected for the new material before it could be incorporated into the SNS target design.

As a first-order generality, cavitation-erosion resistance increases with the surface hardness of the material. Therefore, substantially cold-worked 316LN is considered a candidate replacement for annealed 316LN. In addition, a proprietary surface hardening process called kolsterizing is being investigated as a treatment to improve cavitation-erosion resistance of annealed 316LN. [The kolsterizing process is a low temperature carburization treatment performed by Bodycote Metals Technology of Apeldoorn, Netherlands.] An alternative material, Nitronic 60, is an austenitic stainless steel with somewhat similar composition to 316LN but with a high work hardening rate deemed significant to cavitation-erosion resistance.[10]

Coupons representing these alternatives were exposed along with annealed 316LN in a mercury thermal convection loop (TCL) designed and operated identically to many previous TCLs.[11-15] The purpose of the investigation was to confirm similar compatibility to annealed 316LN in Hg for each alternative or identify the inappropriate alternatives. Another alternate target container material – Inconel 718 – was previously evaluated in a Hg TCL.[12,15]

2. EXPERIMENTAL

2.1 LOOP FABRICATION AND SPECIMEN CHAINS

The TCL used in this experiment was, in fact, used in a prior investigation[13] in which coupons of 316L/316LN were examined in a variety of surface and heat treatment conditions and found to be free of significant interaction with flowing Hg at temperatures up to about 300°C. The TCL was determined to be likewise free of any significant attack and therefore suitable for reuse in this experiment. Only minor welding was required to repair the small cuts in the loop tubing that were made to remove the prior specimen chains and to modify the sealing mechanism on the top of one of the vertical legs.

A schematic of the TCL design is shown in Fig. 1. The TCL was fabricated of mill-annealed 316L SS seamless tubing (25.4 mm OD, 1.8 mm wall) with the composition shown in Table 1. Thermocouple wells, which protruded about a quarter of the diameter into the flow channel, were also seamless, mill annealed 316L SS tubing (6.4 mm OD, 0.7 mm wall). The valves and a few other metallic accessories (connectors, transfer lines, etc.) were also 316 or 316L SS.

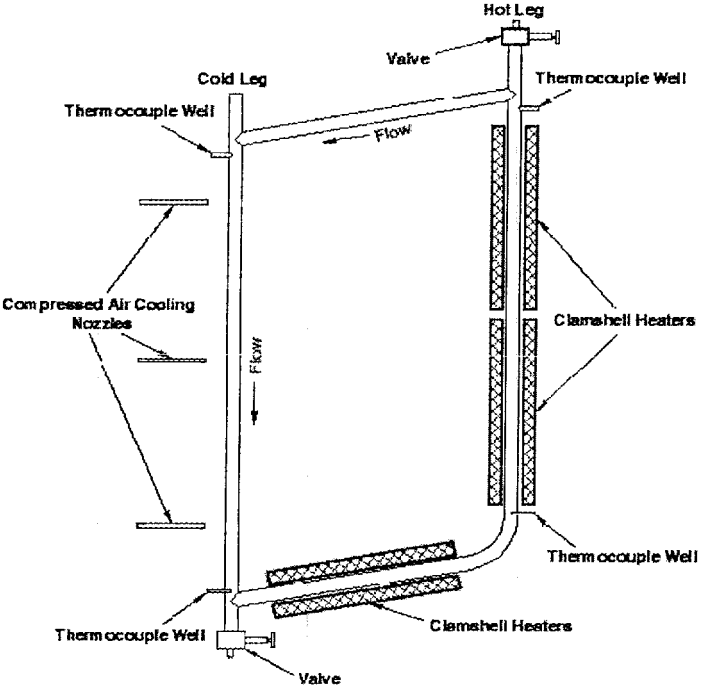

Fig. 1. Schematic of the thermal convection loop design. The distance between thermocouple wells on each vertical section is about 70 cm in the actual loop, and the vertical sections are separated by about 45 cm.

Table 1. Composition of 316L SS TCL tubing and specimens. Data from mill certification for each material, given in weight percent.

Element	316L Tubing	316L Specimens	316N Specimens	Nitronic 60 Specimens
C	0.013	0.018	0.009	0.084
Cb	0.17			
Co			0.16	
Cr	16.75	16.10	16.31	16.15
Cu	0.30	0.29	0.23	0.33
Fe	balance	balance	balance	balance
Mn	1.84	1.73	1.75	7.81
Mo	2.12	2.15	2.07	0.25
N	0.046	0.030	0.11	0.127
Ni	10.19	10.10	10.20	8.15
P	0.028	0.028	0.029	0.024
S	0.014	0.005	0.002	0.016
Si	0.34	0.50	0.39	3.92

Each vertical section of the TCL contained a chain of 32 rectangular coupons of the dimensions indicated in Fig. 2. The specimens were joined together with a continuous 316L SS wire (about 0.4 mm diameter) via the holes in the corners/ends of each specimen. Specimen chains were admitted to the TCL through openings at the top of each vertical leg that were subsequently closed with appropriate high temperature fittings. The end of each wire – which was pulled through a small opening near the bottom of each leg of the TCL – was welded to the bottom of the respective vertical sections to keep the chains from floating to the top of the Hg and positioned such that the top and bottom of the chain corresponded approximately to the thermocouple well positions in each leg. To minimize specimen movement relative to each other and facilitate close spacing, adjacent rectangular coupons were interlocked via the small notch at each end of the specimen; thus, alternating coupons were turned 90° relative to each other.

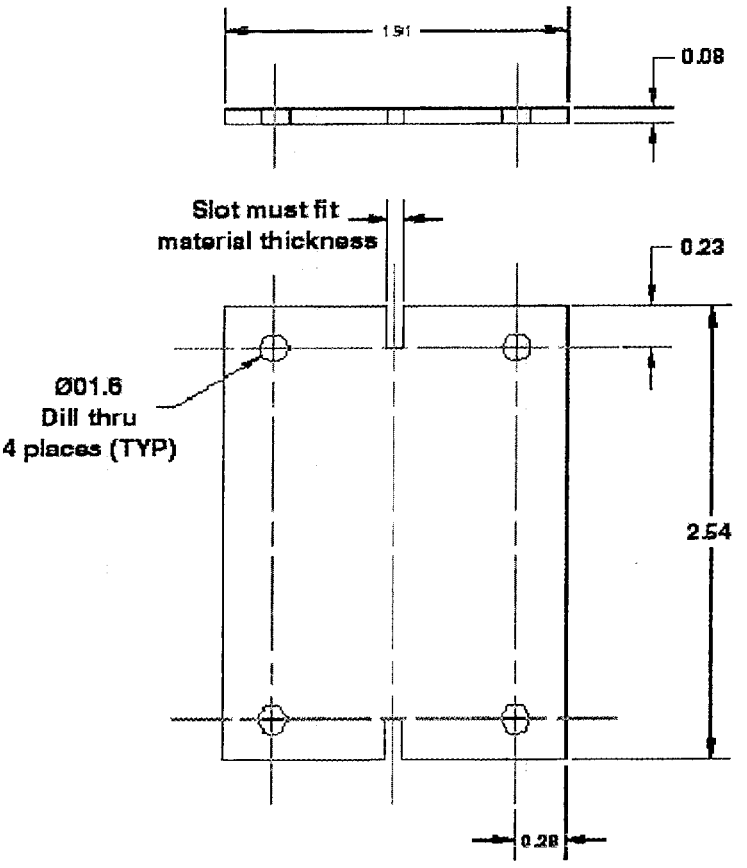

Fig. 2. Dimensions (in cm) of rectangular coupons.

There were several types of coupons exposed in the TCL. All of the 316LN coupons came from the same master heat of 316LN (composition given in Table 1) and were prepared to the same machining specification to generate the base condition coupons. The types of 316LN examined included:

- mill-annealed/surface-ground coupons (approximately 1 μm profile),
- mill-annealed/surface-ground coupons with a bead blasted surface (approximately a 25 μm profile),
- 50% cold-worked/surface-ground (approximately 1 μm profile), and
- mill-annealed/surface-ground (approximately 1 μm profile) followed by a kolsterizing surface treatment to a depth of approximately 33 μm.

Other types of coupons included:

- Nitronic 60 in the mill-annealed/surface-ground condition (approximately 1 μm profile), and

- 316L (not 316LN), mill-annealed/surface-ground followed by a kolsterizing surface treatment to a depth of approximately 33 μm.

The specimens in each chain were individually numbered, cleaned ultrasonically in acetone, and weighed prior to assembly of the chain. All specimens were handled with gloves and tweezers during the stacking and wiring activities. Generally, the specimen chains were assembled with a repeating pattern of coupons – every fourth coupon was mill-annealed/surface-ground 316LN, kolsterized 316LN, 50% cold-worked 316LN, or Nitronic 60. The repeating pattern was interrupted only by one coupon each of kolsterized 316L or bead-blasted 316LN placed near the bottom of each coupon chain.

Prior to original fabrication of the TCL, the ID of the 316L tubing was mechanically and chemically cleaned to remove fabrication debris and make the ID surface as smooth and uniform as possible. Mechanical cleaning of the tube ID was accomplished with a type 302 SS bristle brush attached to an extended rod and powered by a standard hand drill. Subsequently, the tube sections were capped with rubber stoppers and each tube section was filled with a pickling solution (10% nitric acid and 3% hydrofluoric acid in water) for about 10 minutes at room temperature. Following this treatment, the required cutting and bending of the tubes (tubing filled with a very soft Bi-In-alloy to prevent tube collapse) was performed and the tubing was rinsed with alcohol and air-dried. Following the original TCL test, a cotton rag on a wire was forced through the loop tubing to remove residual Hg to facilitate subsequent handling.

Following the weld repairs permitting reuse of the loop, specimen placement, and final assembly, the loops were filled with methanol as a final leak check. Unlike some of the other TCL experiments in this program, no steam treatment was included in the loop preparation.

2.2 FILLING WITH MERCURY

The loop was alternately evacuated (internal pressure of a few microns of mercury) and filled with helium several times. Subsequently, the loop was evacuated and then filled with mercury from the reservoir attached to the top of the hot leg – the ullage of which was also evacuated/purged with helium. A slight positive pressure of helium – about 0.03 MPa (5 psig) – was used as a cover gas for the mercury in the loop. The loop was warmed to near operating temperature and the volume of Hg that expanded above the fill line at room temperature was drained off through a side arm of the loop.

Virgin mercury from the same batch as that used for the original 316L SS loops[11] was used for these experiments. Standard chemical analysis of representative samples

indicated the Hg was quite pure, containing only about 85 ppb Ag and 100 ppb Si above detection limits. Immediately prior to use in the loops, the Hg was filtered through cheesecloth to remove the small amount of residual debris (oxides) floating on the surface of the Hg.

2.3 LOOP OPERATION

Generally, the heat-up and operation of the TCLs was identical to that reported previously[11] for 316L SS TCLs. Clamshell-type furnaces were placed on the vertical leg (two furnaces) and on the near-horizontal lower portion of the loop (one furnace) for long term operation. The control temperature of the lower furnace was kept somewhat below the temperature of those on the vertical hot leg to help maintain the mercury flow pattern. The vertical section of the cold leg was cooled by compressed air delivered from three roughly equi-spaced copper tubes with outlets placed close to the outer loop surface and an array of small fans providing air movement across the entire cold leg. After about a day of furnace temperature and airflow adjustments, the temperature at each thermocouple well and the mercury flow rate became quite stable. About 500 h into the experiment, two brief interruptions due to heater problems were followed by a ~8 h shutdown to replace the suspect heaters. The new heaters were more efficient, and new insulation on the hot leg upgraded the cooling capacity, so a somewhat larger temperature gradient was obtained over the final 1500 h of the experiment. During the experiment, with the exception of the heater problems previously described, the temperature at each location varied only by about ± 2°C. Table 2 compares temperatures at each thermowell for this TCL (before and after the heater repair) with the same data for the original TCL experiment.[11]

The flow rate of the mercury was determined via a localized temperature spike test. In this test, a propane torch was used to heat a small area in the middle of the roughly horizontal section at the top of the loop for about 15 seconds. The time required for the resultant temperature rise to reach each thermocouple in sequence around the loop along with the distance between thermocouples was used to estimate the velocity of the mercury. Based on this technique, the mercury flow rate was found to be about 1.2 m/min with a temperature gradient of 70°C (first 500 h) and about 1.4 m/min with a temperature gradient of 90°C (remainder of experiment).

Table 2. Nominal temperatures at each "corner" of the thermal convection loop described here along with equivalent data for TCL #1 (a previous 316L TCL). Approximately a ± 2°C drift over the duration of the experiment was measured at each location.

	Present experiment		Previous experiment
	First 500 h (°C)	Final 1500 h (°C)	TCL #1 (°C)
Bottom of hot leg	258	252	268
Top of hot leg	294	307	305
Top of cold leg	269	262	280
Bottom of cold leg	224	217	242
Nominal temperature gradient (maximum to minimum)	70	90	63

3. RESULTS AND DISCUSSION

3.1 GENERAL VISUAL ASSESSMENT

The operation of the TCL was terminated after 2000 h operation at the conditions indicated in Table 2. There were a total of three brief periods totaling less than 100 h in which the Hg temperature at each position in the TCL dropped about 60-70°C due to heater problems, but these hours were not counted toward the total of 2000 h operation. In addition, there was one period of about 8 h in which all of the furnaces were turned off and the Hg cooled to near room temperature to facilitate replacement of the heaters. During all of the temperature perturbations, the loop remained closed (no air admitted) and the Hg remained in contact with the specimens.

At the end of the test, the heaters were shut down, the Hg cooled to near room temperature, and the Hg drained from the loop through a valve at the bottom of the cold leg. The Hg appeared clean and shiny with only a trace of oxide-like debris associated with the last few drops to exit the drain valve.

The wires retaining each specimen chain were cut near the weld joint attaching it to the loop tubing and the specimen chains were carefully lifted form the TCLs. Relatively little Hg was observed adhering to the specimens, which is consistent with many previous observations that wetting by Hg – as evidenced by low contact angle and tenacious adherence – tends to diminish dramatically as the temperature is lowered below about 250°C.

Following the TCL test, most of the specimens from each chain were discolored to dark reddish-brown. Curiously, every fourth specimen on each chain – the Nitronic 60 coupons – appeared relatively shiny. Representative photographs depicting this observation appear in Fig. 3.

A large fraction of the discoloration for each of the affected coupons appeared to be superficial – it could be wiped from the coupon surface with a gloved finger or a paper towel. Figure 4 shows a photograph of a coupon after wiping part of the surface with gloved thumb pressure. The reason for the accumulation of this reddish-brown product on certain surfaces and not others is a matter of speculation, but it reinforces the notion that wetting and interaction with surfaces in Hg is a matter associated with the details of surface condition, cleanliness, composition, and other factors influencing surface activity. However, based on the insignificant weight change values (next paragraphs), it would appear that the reddish-brown material did not negatively affect any of the coupons.

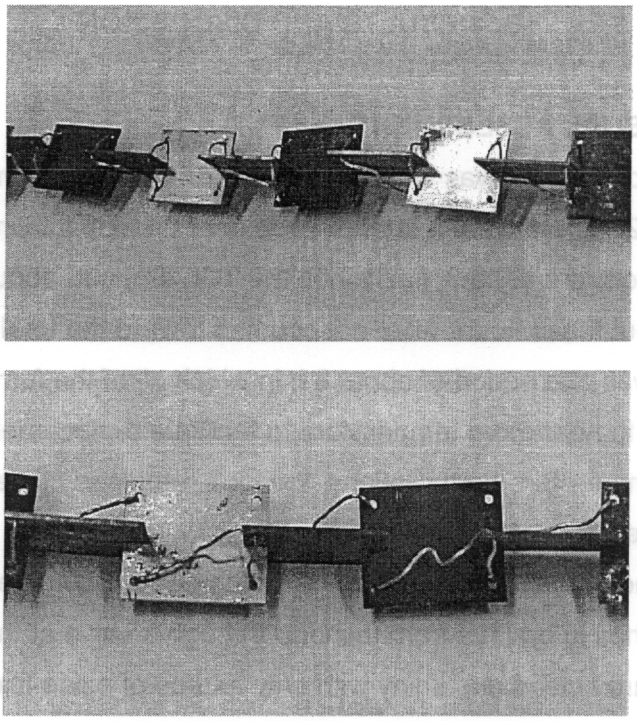

Fig. 3. Post-exposure views of specimen chains. Top: General view of the central section of the hot-leg specimen chain. Bottom: Close-up view of specimens near the top of the cold leg.

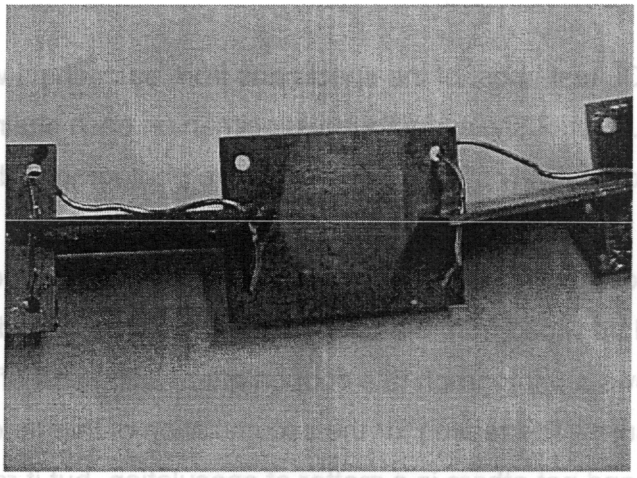

Fig. 4. Reddish-brown film accumulated on many of the specimens can be largely removed by wiping with thumb pressure.

3.2 WEIGHT CHANGE AND METALLOGRAPHY

Following the initial observation gathering, the specimen chains were disassembled and each coupon was rubbed vigorously with HgX (a mercury clean-up cream that binds Hg as a sulfur-bearing compound), rinsed in water, cleaned ultrasonically in acetone, and weighed. Even after this cleaning procedure, many of the coupons retained at least minor brown or dark gray discoloration.

3.2.1 Mill-Annealed 316LN Coupons

The nominal weight change for the mill-annealed/surface-ground 316LN coupons averaged –1.25 ± 0.20 mg, with no apparent trend indicating a weight change dependence on exposure temperature or position on the coupon chain. On a coupon with over 10 cm^2 of surface area, this nominal weight loss corresponds to a negligible uniform attack (less than 1 μm/y). Post-test metallographic cross-sections of the annealed 316LN coupons revealed no change in surface roughness or any indication of localized attack. This behavior is consistent with that observed for 316L/316LN in several other TCL experiments in this program, with the minor exception that some of the 316L coupons in the initial TCL experiment[11] exhibited more weight loss (corresponding to a maximum of about 15 μm/y) as well as Ni and Cr leaching from the surface.

Each coupon chain included one 316LN mill-annealed specimen with a bead-blasted finish. Each of the bead-blasted 316LN specimens exhibited a weight loss of about 0.50 mg. It is difficult to assign any practical significance to the smaller weight loss for bead-blasted coupons compared to surface-ground coupons, but perhaps the type/extent of deformation generated by bead-blasting (20-25 μm profile) made wetting by Hg even more difficult than for the nominal surface condition (~1 μm profile). Figure 5 shows a cross-section of a bead-blasted 316LN coupon following exposure in this TCL test. The extent of surface roughening observed here is about 20 μm and unchanged from the initial exposure condition.

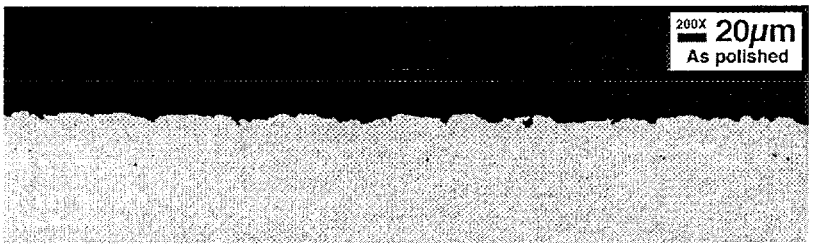

Fig. 5. Surface of 316LN coupon which was bead-blasted. The extent of surface roughness for this coupon exposed 2000 h at about 260°C is unchanged from the initial condition.

3.2.2 Cold-Worked 316LN Coupons

The nominal weight change for the 50% cold-worked/surface-ground 316LN specimens averaged –0.37 ± 0.15 mg. Like the mill-annealed/surface-ground specimens, no weight loss trend associated with coupon exposure temperature or position around the loop was identified. Further, no attack or change in surface roughness as a result of exposure was observed. The hardness increase from the mill-annealed condition (HRB 55-60) to the 50% cold-worked condition (HRC 30-35) was significant, but it is not possible to determine from the data if the hardness increase was directly responsible for the decrease in weight loss for these coupons. Indirectly, the rolling process used to cold work the specimens and the slightly different handling/machining may have contributed to a subtle difference in surface chemistry/reactivity that influences interaction with Hg.

3.2.3 Mill-Annealed Nitronic 60 Coupons

The nominal weight change for the mill-annealed/surface-ground Nitronic 60 specimens averaged –0.36 ± 0.18 mg. Again, it is difficult to assign significance to such low values of weight loss, but this number is less than the annealed 316LN and essentially identical to that for the 50% cold-worked 316LN. No indication of attack or increase in surface roughness as a result of exposure was observed (see Fig. 6). The hardness of the mill-annealed/surface-ground Nitronic 60 was HRB 81-86, which is somewhat greater than the equivalent value for the mill-annealed/surface-ground 316LN. In this case, the difference in bulk chemistry is likely to be more significant than the small hardness difference between annealed 316LN and annealed Nitronic 60 in terms of potential interaction/reactivity with Hg.

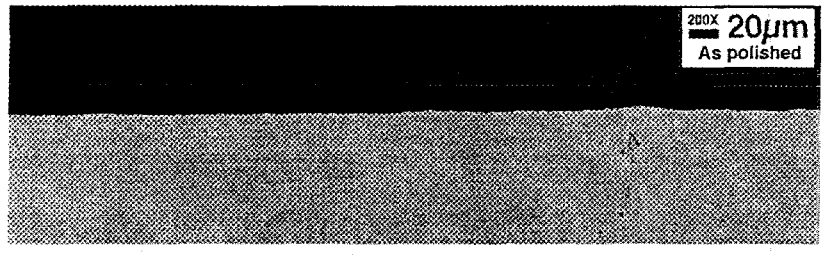

Fig. 6. Representative surface of a Nitronic 60 coupon exposed for 2000 h at about 305°C. The extent of surface roughness for this coupon is unchanged from the initial condition.

3.2.4 Kolsterized 316LN and 316L Coupons

The specimens for kolsterizing were to receive the "33 μm treatment," meaning generically that the surface was impregnated with solid solution carbon such that some property (hardness, %C, etc.) is changed in a measurable way to a nominal or practical depth of about 33 μm. Due to the proprietary nature of the process, no details regarding the %C as a function of depth into the material were provided by the supplier. However, there is no reason to assume that the impregnation of carbon ceases at a depth of 33 μm but rather that the maximum or average effect is between the surface and a depth of 33 μm. While hardness measurements were not made directly on the coupons used in this experiment, measurements on similarly kolsterized coupons for cavitation studies[7] indicated a surface microhardness on the order of 1000 DPH (HRC 70).

The kolsterized specimens were non-uniformly discolored brownish to dark gray following the carburizing treatment and, as described previously, were uniformly dark reddish-brown following the TCL test. The nominal weight loss for the kolsterized specimens exhibits more scatter than for the other specimen types in this experiment, but the nominal weight change for 316LN specimens exposed in the cold leg (–1.16 ± 0.20 mg) was slightly greater than for equivalent specimens exposed in the hot leg (–0.91 ± 0.16 mg). Of course, in a practical sense, this difference is inconsequential to the operation of the SNS, because both values correspond to a negligible extent of attack over any expected service life.

Examination of the weight change data for the kolsterizing process itself indicated that the fifteen 316LN specimens (one of the original sixteen was discarded as an obvious outlier) exhibited a weight gain – presumably resulting from carbon doped into the surface – of 2.56 ± 0.16 mg. Using the area of the coupon (~10 cm^2) and assuming the weight gain is uniform from the surface to a depth of 33 μm, it is possible to estimate

the average amount of carbon in the surface layer to be about 1% (by weight) of carbon to a depth of 33 μm, and a much higher concentration is likely in the outer few microns of the surface.

Only four 316L specimens – machined, sized, and prepared to the same specification as the 316LN specimens – were also kolsterized in the same batch treatment. It is potentially significant that the nominal weight gain resulting from the kolsterizing process for these four 316L specimens was 3.77 ± 0.06 mg, which is 47% greater than for the equivalent 316LN specimens. This result suggests that the kolsterizing process may be very sensitive to base composition – the high nitrogen content of the 316LN material can change the solubility or kinetics of the process such that 316LN was able to accept significantly less carbon.

Both of the kolsterized 316L specimens that were exposed in the TCL (one in each leg) experienced a weight change of –0.45 mg, or about half that of the kolsterized 316LN coupons. Again, the overall weight change is too small to have practical significance in either case. It is perhaps significant, however, that the greater extent of carburization in 316L (based on weight change as a result of the process) is also associated with lower weight loss in the Hg TCL.

In a separate SNS experiment, buttons for cavitation-erosion testing in Hg using the vibratory horn[8, 16] were also kolsterized. All five of the buttons were 316LN of the same composition given in Table 1, but three were in the as-machined state (had lathe rings and a slight surface profile) and two were polished on 600-grit paper (macroscopically smooth). There was very little scatter in the weight gains resulting from the process, and the polished specimens gained 2.5 times the mass of the rougher machined specimens in a single kolsterizing treatment (to 47 μm in this case). Combined with the aforementioned result, the difference in weight gains for various specimens indicates the kolsterizing process is very surface/alloy sensitive. While that is not a surprise for a diffusion process, the magnitude of the sensitivity suggests that it must be better understood prior to preparation of specifications for SNS service that include the kolsterizing process.

4. CONCLUSIONS

The results of this TCL experiment indicate that mill-annealed 316LN and variants which include 50% cold work, a kolsterizing surface treatment, and a composition modification (Nitronic 60) all exhibit similar acceptable compatibility with Hg. Suitable compatibility was evidenced by inconsequentially low weight losses and no metallographic evidence of attack or increase in surface roughness. Weight changes associated with the kolsterizing treatment itself indicate a process that is reproducible but apparently very sensitive to base composition and/or surface condition. This latter observation suggests due care is necessary in specifying the process to obtain particular levels of carbon impregnation or specific mechanical property results.

5. ACKNOWLEDGMENTS

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