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CORROSION OF ALLOY 718 IN A MERCURY THERMAL CONVECTION LOOP

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Metals and Ceramics Division

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CONTENTS

TABLES

FIGURES

CORROSION OF ALLOY 718 IN A MERCURY THERMAL CONVECTION LOOP*

S. J. Pawel, J. R. DiStefano, and E. T. Manneschmidt

ABSTRACT

Two thermal convection loops (TCLs) fabricated from annealed alloy 718 continuously circulated mercury (Hg) and Hg with 1000 wppm gallium (Ga), respectively, for about 5000 h, duplicating previous TCL tests for annealed 316L. In each case, the maximum loop temperature was 305°C, the minimum temperature was 242°C, and the Hg flow rate was approximately 1.2 m/min. Unlike the 316L exposed to Hg, which above about 260°C exhibited a thin, porous surface layer depleted in Ni and Cr, the alloy 718 coupons revealed essentially no wetting and, therefore, no interaction with the Hg at any temperature. Alloy 718 coupons suspended in the loops revealed inconsequentially small weight changes, and both the coupons and loop tubing exhibited no detectable metallographic evidence of attack.

*Research sponsored by the U. S. Department of Energy for the Spallation Neutron Source.

1.0 INTRODUCTION

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

The nickel-base alloy 718 is under consideration as a possible alternate target containment material. Compared to 316L/316LN SS, alloy 718 is considerably stronger and therefore potentially offers reduced section thicknesses (for improved heat transfer) or larger factors of safety for equivalent section thicknesses. In addition, some positive experience with alloy 718 as a window material in spallation systems has been accumulated.[2] However, previous corrosion results[3] with thermal convection loops (TCLs) indicated that Ni (and to a lesser extent Cr) can be preferentially leached from 316L SS by hot, flowing Hg. Since alloy 718 generically contains five times as much nickel as 316L SS, the corrosion resistance of alloy 718 for service as Hg containment has been questioned. In other liquid metal or molten salt systems in which preferential leaching of Ni is observed (for example, lithium[4,5] and lithium hydride[6]) corrosion/dissolution rates increase with increased Ni content in the alloy.

In this study, corrosion of alloy 718 in Hg was examined in TCLs using procedures essentially identical to those previously used to examine the interaction of 316L SS with Hg.[3] As before, the TCLs were operated for about 5000 h at temperatures somewhat higher than those expected in the actual SNS target in order to encourage chemical wetting and therefore exacerbate corrosion. As before, "twin" TCLs were operated: one containing pure Hg as the working fluid and one with 1000 wppm Ga added to the Hg (as a potential aid to wetting).

2.0 EXPERIMENTAL

2.1 LOOP FABRICATION

A schematic of the TCL design is shown in Fig. 1. Each TCL in this study was fabricated of mill annealed alloy 718 seamless tubing (25.4 mm ID, 1.8 mm wall) with the composition shown in Table 1. The thermocouple wells, which protruded about halfway into the flow channel, were also seamless, mill annealed 718 tubing (6.4 mm OD, 0.7 mm wall). The valves and a few other metallic accessories (connectors, transfer lines, etc.) were 316L SS. [This design is identical to previous TCL tests with 316L,[3] except that the present loop design used 25.4 mm ID tubing rather than 25.4 mm OD tubing - thereby requiring a slightly larger Hg inventory.]

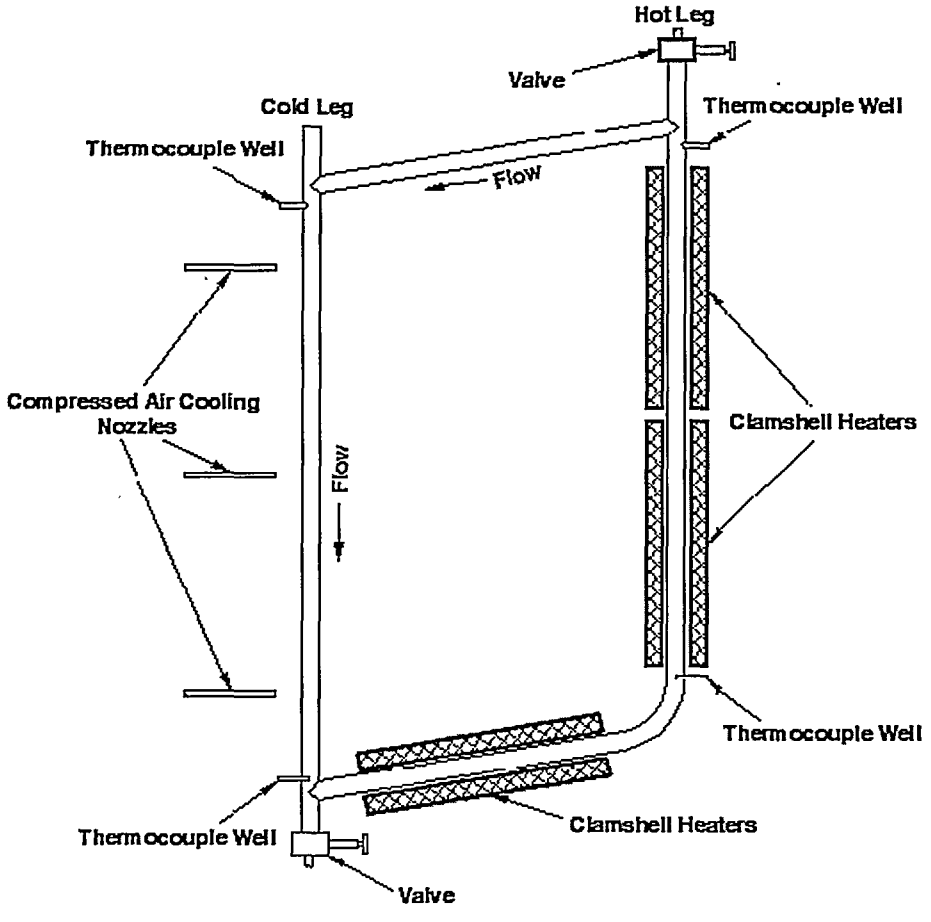

Fig. 1. Schematic of the thermal convection loop design. The distance between thermocouple wells on each vertical section is about 70 cm in the actual loop, and the vertical sections are separated by about 45 cm.

The vertical portions of each TCL contained a chain of alloy 718 specimens. Each specimen chain consisted of 30 rectangular coupons and 2 miniature tensile specimens (see Fig. 2 for specimen dimensions) joined together with a continuous alloy 718 wire (about 0.4 mm diameter) via the holes in the corners/ends of each specimen. The end of each wire was welded to the bottom of the respective vertical sections to keep the chains from floating to the top of the Hg. 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.

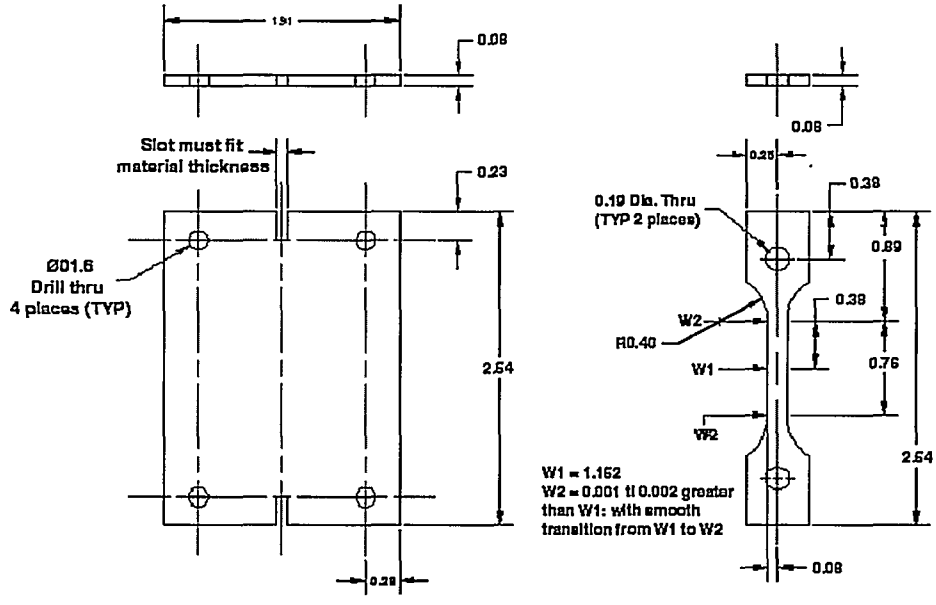

Fig. 2. Dimensions (in cm) of rectangular coupons and miniature tensile specimens.

All of these specimens were prepared from the same heat of material, and the composition is given in Table 1. All of the rectangular coupons were identical (surface ground finish) except for two in each chain (positions 2 and 31, with position 1 at the top), which were polished on one of their large faces through 1 um alumina paste to examine any potential role of surface finish. Miniature tensile specimens, included at positions 5 and 28 in the chain, also had a surface ground finish. Figure 3 shows a portion of a specimen chain indicating the arrangement and relative polish of the specimens.

Table 1. Composition of alloy 718 TCL tubing and specimens. Data from mill certification for each material, given in weight percent.

Element	Tubing	Specimens
Al	0.56	0.54
B	0.003	0.004
C	0.03	0.050
Co	0.13	0.40
Cr	18.39	18.13
Cu	0.05	0.05
Fe	18.52	18.35
Mn	0.06	0.21
Mo	2.95	3.01
Nb	5.09	5.07
Ni	53.14	52.70
P	0.010	<0.005
S	0.001	<0.002
Si	0.12	0.13
Ta	-----	<0.005
Ti	0.95	1.06

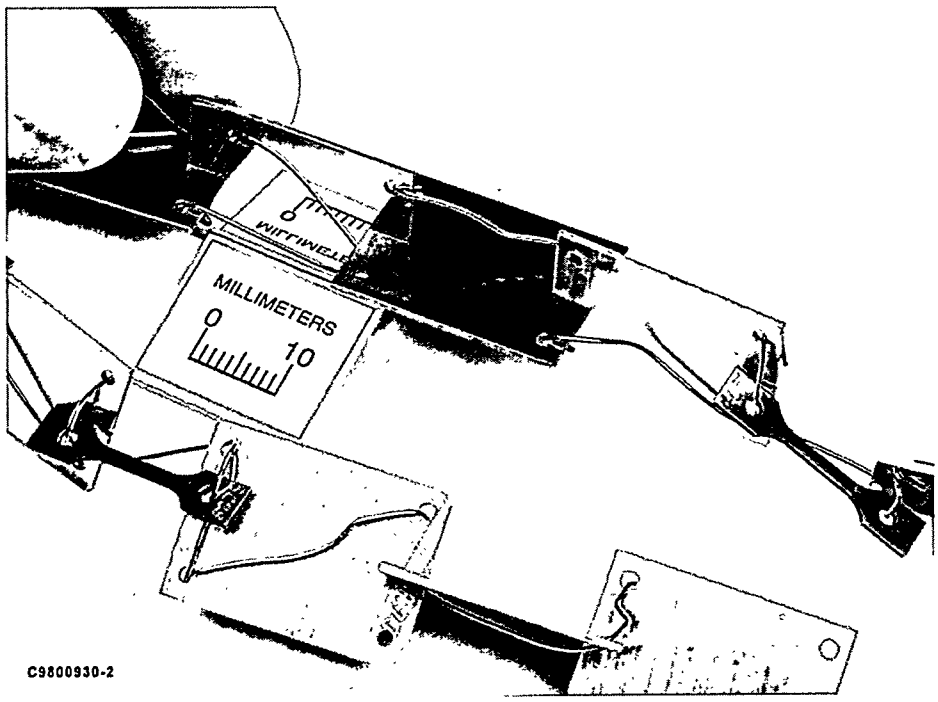

Fig. 3. Section of the alloy 718 specimen chains. Note the polished face of the specimen bearing the reflection of the scale marker. Note that most specimen surfaces were not polished.

The specimens were individually numbered, cleaned ultrasonically in acetone, and weighed prior to assembly of the specimen chain. All specimens and wires were handled with gloves and tweezers during the interlocking and wiring activities. Once in place, the specimen chain extended the entire length of each vertical leg (between the thermocouple well positions). Prior to fabrication of the TCLs, the ID of the alloy 718 tubing was mechanically and chemically cleaned to remove as much oxide/scale as possible. Mechanical cleaning of the tube ID was accomplished with a 302 SS bristle brush attached to an extended rod and powered by a standard hand drill. Subsequently, a pickling solution (16 parts water, 10 parts reagent grade nitric acid, 1 part reagent grade hydrofluoric acid, ambient) was prepared and the tube filled for an approximately 30-minute soak in mostly stagnant solution. [It was found that occasional swabbing with cotton greatly increased the effectiveness of the pickling solution.] Prior to the mechanical/chemical treatment, the tube ID exhibited a dark gray matte appearance. After the treatment, the tube ID had a silver appearance with some slight luster.

Following fabrication and specimen placement, the loops were filled with methanol as a final leak check of the assembly. Unlike the situation for the previous 316L TCLs, no steam treatment was included in the loop preparation.[3]

2.2 FILLING WITH MERCURY

Virgin mercury from the same batch as that used for the 316L SS loops[3] 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.

The procedure for filling of the loops with Hg or Hg+1000 wppm Ga was exactly as described for the 316L SS loops.[3] As before, residual helium (high purity) was the cover gas for the Hg inside the loops.

2.3 LOOP OPERATION

The alloy 718 loops were operated as described previously for the 316L loops.[3] Each loop (Hg and Hg+1000 wppm Ga) was operated for 4950 h at the conditions indicated in Table 2 with only one overnight power outage for each. As before, a temperature-spike test was used to

determine the Hg flow rate in each loop and it was found to be very consistent at 1.1 to 1.2 m/min throughout the duration of the tests.

Table 2. Nominal temperatures at each "corner" of the 718 TCLs compared to the equivalent values for the 316L TCLs. Continuous strip chart print-out of temperatures for each thermocouple over the duration of the 718 experiments indicates a +/- 1°C variation at all positions except the top of the cold leg (+/- 3°C). Nominal temperature gradient in each case = 63°C.

	718 loops	316L loops
Bottom of hot leg	259°C	268°C
Top of hot leg	305°C	305°C
Top of cold leg	284°C	280°C
Bottom of cold leg	242°C	242°C

3.0 RESULTS

3.1 Hg LOOP

Following operation, and after about an hour of cooling, the working fluid was drained from each loop. The pure Hg remained bright and shiny with no visual indication of contamination.

With a minimum of jostling/vibration, the specimen chains were carefully removed from each loop. In the case of the pure Hg loop, the specimens were essentially devoid of any indication of wetting or other interaction with the Hg. The only exceptions were some spots of light brown staining primarily confined to the polished coupons. [See Fig. 4.] Post-test cleaning, which included light wiping with cheesecloth and an ultrasonic soak in acetone, had no effect on the apparent stains.

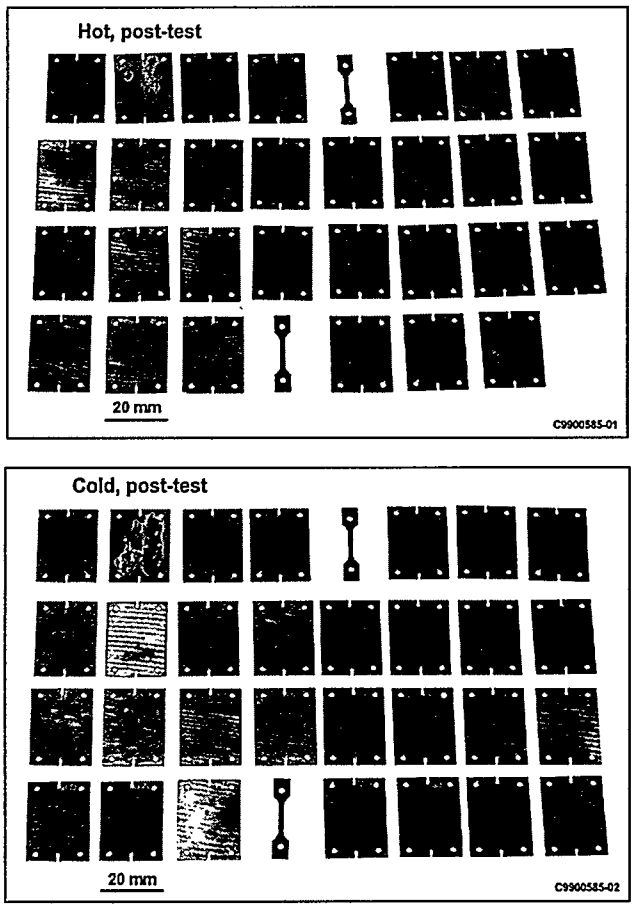

Fig. 4. Post-test appearance of specimens removed from the hot leg (top) and cold leg (bottom) of the Hg loop. Coupons 1-8 in top row, 9-16 in second row, etc.

In the Hg loop, no specimens gained weight and the maximum weight loss among the flat coupons was 0.40 mg (hot leg, position 6), which corresponds to a uniform thickness loss of less than 0.1 μm in almost 5000 h. [For comparison, the maximum weight loss in the corresponding coupon exposures of 316L was 16.5 mg, equivalent to about 3.5 μm of uniform attack.] The next highest weight losses were about 0.20 mg, exhibited by a few coupons at seemingly random locations in either the hot leg or cold leg (no obvious correlation to coupon location/temperature). Most coupons exhibited a weight loss of less than 0.15 mg, and the precise magnitude of weight change did not appear to correlate to coupon location/temperature in any way. The polished coupons all exhibited a weight loss of 0.05 mg or less.

Interestingly, the weight loss per unit area was significantly higher for the miniature tensile specimens than for the rectangular coupons. The maximum weight loss for a tensile specimen, 0.89 mg, occurred in the hot leg at position 5 (adjacent to the rectangular coupon with the greatest weight loss). However, note that the rectangular coupon has about five times the exposed surface area, meaning that for this particular tensile specimen, the weight loss per unit area was about eleven times greater than the adjacent rectangular coupon with maximum weight loss. The weight loss per unit area of the other tensile specimens was similarly larger than for either of the adjacent rectangular coupons.

The miniature tensile specimens were pulled to failure at room temperature at a constant crosshead speed of 0.84 mm/min. Specimens exposed in the loop were tested in the same batch as unexposed specimens and, within the typical scatter of the technique, no differences in mechanical properties were detected between exposed and unexposed specimens. The mechanical properties measured in this way indicated that the sheet stock from which the rectangular coupons and tensile specimens were fabricated was in the mill-annealed condition. [It was anticipated that this material would be in the aged condition, as it was intended to test aged material (coupons) and annealed material (tubing walls) in the same test. However, subsequent hardness tests and a more complete paper trail confirmed the mill-annealed nature of the specimens.]

Representative coupons, portions of the tensile specimens, and ring-shaped segments of the loop tubing were mounted in cross section for standard metallographic preparation. No indication of corrosion or leaching of any kind was detected. In fact, compared with cross sections of unexposed specimens of coupons, tubing, and tensile specimens, there was also no indication of a change in surface roughness on the exposed surfaces.

3.2 Hg+Ga LOOP

When the Hg+Ga loop was drained immediately after the test, the mixture was initially very shiny but, as expected, rapidly formed a gray scum upon exposure to air (a reaction with dissolved Ga).

Unlike the result for the Hg loop, some residual liquid metal was observed on most of the specimens immediately following removal of the specimen chain from the loop. Representative photographs are shown in Fig. 5. Close inspection shows that, at least after brief exposure to air, the residual material is clearly not wetting the specimen surface. Rather, the residual material appears to be a "film" with a large contact angle at the edges that is peeling and curling from the specimen surface. Subsequent handling and cleaning revealed that the film indeed had little or no adhesion and that the original machining marks were still clearly visible beneath the film (indicating little or no interaction with the specimen).

Overall, the weight change for coupons exposed in the Hg+Ga mixture was less than for the specimens exposed to pure Hg. Only a small number of specimens lost weight during the test, including all four of the miniature tensile specimens (maximum loss was 0.25 mg, position 5 in the cold leg) as well as three rectangular coupons (maximum loss 0.06 mg, position 6 in the hot leg). Most of the coupons gained a small amount of weight up to a maximum of 0.23 mg (position 22 in the hot leg). All of the polished specimens gained weight (up to a maximum of 0.22 mg at position 31 of the cold leg).

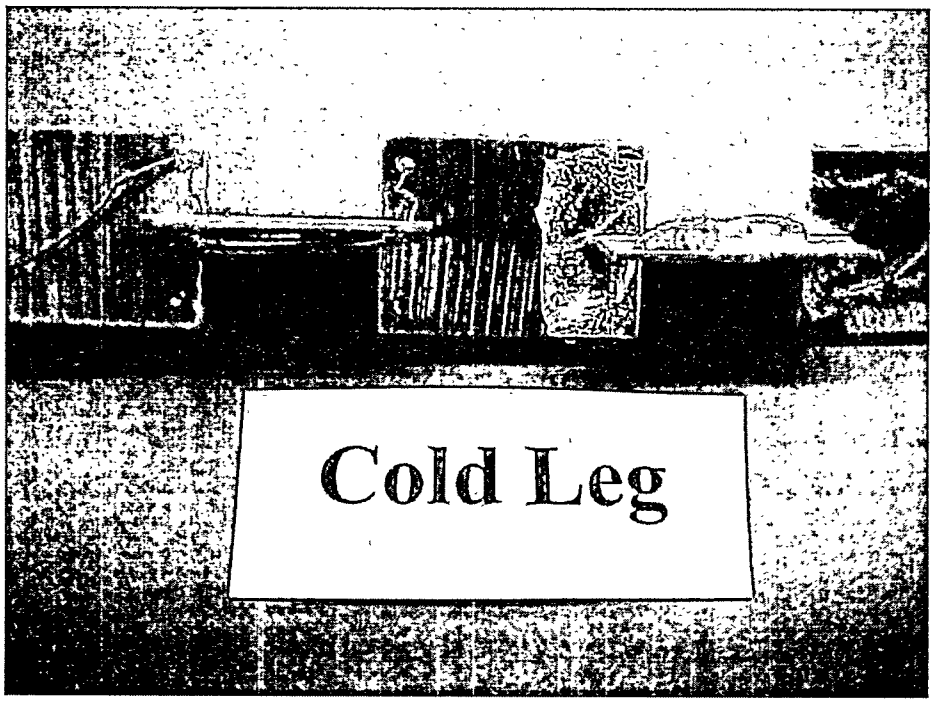

Fig. 5. Representative post-test appearance of specimens exposed in the Hg + Ga loop. Note the "curling" of the film at the edges.

4.0 DISCUSSION

In previous loop tests, exposure of 316L to Hg under conditions essentially identical to those for the alloy 718 loops resulted in leaching of Ni, and to a lesser extent Cr, from the surface of the 316L coupon specimens and the concomitant development of a ferritic, porous surface layer. Since alloy 718 contains approximately five times as much Ni as 316L, it was expected that alloy 718 would reveal a greater extent of attack/leaching than 316L for equivalent exposures in Hg. However, the results for the present tests indicate only inconsequentially small weight changes and no detectable metallographic evidence of attack for the alloy 718 in either Hg or Hg with 1000 wppm Ga.

One explanation for the absence of attack on the alloy 718 tube/coupons could have been that the Hg used for the test was already saturated with components of 718 that are soluble in Hg at the TCL operating temperatures. [Such saturation would not influence temperature gradient mass transfer, but it would eliminate the dissolution required to achieve the initial saturation.] However, virgin Hg (with documented compositional analysis) was used for these tests. Further, due to the fact that the tube ID was slightly larger for the alloy 718 loops than for the 316L loops, the total volume of Hg available for dissolution reactions was actually slightly larger for the alloy 718 experiments. [The ratio of total surface area of tubing plus coupons to volume Hg was slightly lower for the 316L experiments.]

Based on the results obtained to date (tests described in Ref. 3 and those described here), it would appear that - for the same operating conditions at temperatures to about 300°C - alloy 718 is more resistant to wetting by Hg than is 316L. One other observation has been reported indicating potentially increased resistance to wetting by Hg for alloy 718 compared to 316L. In preliminary tests of temperature gradient mass transfer in mercury,[7] specimens of 316 and alloy 718 were exposed to Hg in "rocker tests" for up to 2800 h. In the only exposure for which direct comparison of compatibility response for 316 and alloy 718 is possible, the environment was Hg with 100 wppm Ga cycled between about 350°C (hot zone) and about 270°C (cold zone) for 2800 h. Comparing hot zone specimens, the 316 specimen showed a modest weight loss while the alloy 718 specimen showed a slight weight gain. Little additional analysis was undertaken,[7] but the trend was the same: even at 350°C, alloy 718 was more resistant to interaction with Hg than the 316.

Although the mechanism for increased resistance to wetting for alloy 718 can not be precisely defined, combinations of several factors, including bulk composition of the metallic substrate (and the passive film thereon), the surface condition of the material under investigation, and environmental factors associated with the test itself, may contribute. These possibilities are reviewed in the paragraphs that follow.

As indicated in Table 3, the 316L and alloy 718 used in the loop experiments are quite different with respect to many major alloying elements. Without similar tests on a variety of materials with well-defined compositions, it is not possible to assign increased wetting resistance to any particular element or combination of elements for such widely differing materials. Further, to the author's knowledge, the role of a specific component of the bulk composition on wetting/dissolution of engineering materials exposed to Hg has not been rigorously assessed. However, there are examples in the literature of significant differences in tensile/fracture behavior in Hg between materials for which the bulk composition difference is more minor or confined essentially to single elements. For example, Krupowicz[8] reported the relative resistance of several austenitic stainless steels to liquid metal embrittlement (LME) in room temperature Hg. The investigation utilized slow strain rate tests and compared the post-test reduction in area (RA) of specimens tested in air and in Hg. It was found that for as-received (solution annealed and cold straightened) material, 304 and 304L stainless steel were somewhat susceptible to LME in Hg as indicated by a substantial decrease in RA (from in excess of 80% in air to as low as 35% in Hg) and the formation of many secondary cracks. However, specimens of 316 and 316L were essentially immune to LME in Hg under these conditions, as evidenced by a decrease of only 0-2% in RA and the absence of secondary cracks. Other than minor variations in Ni and Cr, the major compositional difference between these alloys is that the 316/316L alloys (no LME in Hg) contain >2% Mo where the 304/304L alloys (susceptible to LME in Ref. 8) contain no Mo. [Interestingly, the presence of Mo in 316/316L is known to impart increased stability to the passive film compared to that formed on 304/304L such that aqueous corrosion resistance (general and localized) is often significantly enhanced.] Krupowicz[8] also included type 321 stainless steel (Fe17Cr10Ni0.3Mo0.6Ti) as well as alloy 600 (Ni15Cr10Fe) and alloy 800 (Fe31Ni22Cr0.4Ti) in the test matrix. In the annealed condition, the high nickel alloys exhibited intermediate degradation of RA in Hg while type 321 performed similarly to type 316/316L.

Table 3. Composition comparison for major alloying elements between 316L coupons (used in previous experiments[3]) and alloy 718 coupons used in the present experiments. Compositions given in weight percent.

	316L SS	Alloy 718
Cr	16.1	18.1
Cu	0.3	0.1
Fe	69.0	18.3
Mo	2.1	3.0
Ni	10.1	52.7
Nb	0.0	5.1
Ti	0.0	1.1

Clearly, Hg cannot have an influence on material properties unless the Hg interacts with (chemically wets) the material, and the literature results discussed here suggest that even minor variations in bulk composition potentially influence such interaction significantly during a dynamic (active plastic strain) test. In the particular case of 304 vs 316 [ref. 8], the bulk Mo content appears to be a significant variable. However, Mo would not be expected to have the same effect in every alloy family or the same effect in a mechanically static test (such as the TCLs described here). Further, wetting is no doubt a very surface sensitive phenomenon, so local surface chemistry may be a significant factor as well. [To the authors' knowledge, this factor has not been reported in the literature for Hg.] Therefore, the results of any compatibility test will be difficult to predict based only on the bulk composition of the test material.

The surface condition of the test material is also potentially an important factor. In the mechanically-static TCL tests, the annealed alloy 718 was essentially unaffected by Hg in several conditions: (a) surface ground coupons, (b) polished (mirror-finish) coupons, and (c) tubing that was mechanically brushed and chemically pickled. In terms of weight loss, the most significant change was for the miniature tensile specimens, which were surface ground over most of the exposed surface but the edges of the specimens were cut with an electro-discharge machining technique that left a relatively rough (higher surface relief) finish. Potentially, some feature of this edge surface condition contributed to the relatively higher weight loss per unit area of the miniature tensile specimens, but the overall effect was still very minor.

In the 316L TCL tests,[3] only the coupons (not the surrounding tubing) exposed to Hg near 300°C revealed any significant interaction. It is not clear why the tubing might be unaffected since it was a heat transfer surface and, as such, was slightly hotter than the coupons. However, the 316L tubing surface did not receive any initial mechanical cleaning nor did it receive any pre-exposure pickling. In contrast, the coupons were surface ground, and both the tubing and coupons received a steam treatment prior to initiation of the test. Again, these results point to the importance of surface condition as one variable affecting corrosion in Hg.

The contact angle generated by drops of Hg on any given surface (an indication of wetting) is critically dependent on the surface layer "cleanliness" as opposed to any bulk properties.[9] In particular, the presence of adsorbed impurities in amounts even less than a full monolayer of coverage was found to significantly alter apparent wettability. Wilkinson[9] cited work indicating specimens of Fe, Ni, Mo, W, and Ti that were polished, degreased, and bombarded with argon ions under vacuum immediately prior to Hg drop placement on the specimens showed similarly high contact angles (poor wetting) compared with surfaces for which little cleaning effort was expended. However, the contact angles decreased to zero (complete wetting) when the argon ion bombardment was reinitiated after Hg drop placement. The conclusion was that contamination of the surface could happen very quickly and wetting occurred only if the surface was completely free of contaminants (in this case, when cleaned in the presence of Hg).

Lending some credibility to the inhibiting effect of residual oxide films is the observation that small additions of Ti and/or Mg to Hg have been shown to improve heat transfer between Hg and steel at relatively high temperatures.[10] The mechanism by which this occurs has not been exhaustively studied, but it appears that Mg and Ti are capable of "gettering" oxygen from both the Hg and from the steel surface, thus eliminating oxide-films on steel thereby improving wetting. Clearly, the addition of Ga to Hg had no such effect on either 316LSS or alloy 718. Although pure Ga readily alloys with many elements, it is not a particularly strong oxide former and, thus, had little apparent effect in promoting wetting by Hg.

5.0 CONCLUSIONS

Two TCLs fabricated with annealed alloy 718 were operated continuously for about 5000 h under conditions duplicating previous tests for 316L (tests with Hg or Hg with 1000 wppm Ga, maximum temperature 305°C, minimum temperature 242°C, fluid velocity 1.2 m/min). In the previous tests, 316L developed a thin porous layer, significantly depleted of Ni and Cr, on the surfaces exposed to Hg above about 260°C. Alloy 718, containing five times as much Ni and slightly more Cr than 316L, showed insignificant weight changes, no evidence of microstructural attack, and no evidence of wetting at all exposure temperatures. The reasons for the absence of wetting in alloy 718 compared with 316L are not clear, but elements of the bulk composition other than Ni and Cr (such as Mo) are potential factors. In addition, subtleties associated with material surface condition and surface cleanliness may be factors. Based on these results, alloy 718 appears suitable as an alternate target containment material; however, more prototypic tests in which wetting is encouraged and dynamic mechanical loads are included, should be considered prior to a final conclusion.

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