MATERIALS COMPATIBILITY TESTING FOR A PILOT-SCALE OXIDE REDUCTION SYSTEM

by

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Abstract

Materials compatibility tests were performed on five candidate steels for the pilot-scale oxide reduction system currently under development at Argonne National Laboratory – West. Three stainless steels (types 316L, 304, 347) and two alloy steels (2.25Cr-1Mo and 9Cr-1Mo-V) were evaluated. Small, Swagelok-sealed capsules constructed from each steel were loaded with a LiCl salt solution and exposed at 725°C for 30, 60, and 90 days. The LiCl salt solution contained 3.5 wt% Li₂O, 1 wt% Li₃N, and was saturated with Li metal. The extent of corrosion was assessed following exposure by destructive metallographic examination of each capsule. The extent of corrosion observed varied widely, from essentially none to full penetration of the capsule wall. The primary form of corrosion for all steels was grain boundary porosity and depletion of Cr from the exposed surface.
Introduction

Argonne National Laboratory (ANL) is developing an oxide reduction system to be used in conjunction with its electrometallurgical (EM) treatment process for DOE spent nuclear fuel (SNF). The oxide reduction system will pre-condition spent oxide fuel, transforming uranium and transuranic constituents of the fuel into metals prior to electrorefining in the EM process. Reduction is accomplished at 650°C in a pool of molten LiCl salt saturated with Li metal. The reduction process has been demonstrated at lab and engineering scales [1]; a pilot-scale oxide reduction (PSOR) system is under development [2].

An important aspect of the PSOR system development is material selection. Efforts to date have focused on the reduction vessel, and the nominal conditions mentioned above represent an aggressive corrosion environment. In addition, impurities in hot cell gas environment in which the PSOR reduction vessel will operate are expected to enhance corrosion. Combined oxygen and moisture levels in the nominally Ar gas are controlled to 60±40 ppm, while nitrogen levels can approach 10,000 ppm [2]. These gaseous impurities may interact with the molten LiCl/Li solution to form oxide and nitrides and accelerate corrosion of the vessel materials.

Corrosion of structural materials in the reduction vessel environment has not been extensively studied, particularly the combination of LiCl molten salt and liquid Li metal. The corrosion of metals by liquid Li has been studied fairly extensively [3,4]. Ferrous alloys perform fairly well, with preferential dissolution of Ni as the principal form of attack. The level of nitrogen impurity in the Li strongly influences the corrosion rate [4]. Less attention has been paid to corrosion by molten LiCl salts [5], but the available literature indicates that the primary mechanism of attack in ferrous alloys is preferential dissolution of Cr.

Two studies have been performed to assess the corrosion of candidate PSOR materials in the reduction vessel environment. Olson, et al. [6] tested a number of refractory alloys in a LiCl-Li2O-Li3N-Li molten salt solution at 725°C. The alloys were immersed in the solution in an open crucible contained in a nominally Ar glovebox. Severe corrosion of the alloys was reported, which was attributed to oxygen contamination of the salt solution from the glovebox atmosphere or the ceramic crucible material. Indacochea, et al. [7] tested several ferrous alloys and tantalum in a similar salt solution, again contained in a ceramic crucible in a glovebox. Contrary to the previous study, no corrosion of any of the materials was detected after thirty days of exposure. The level of oxygen impurity was tightly controlled in this study. In a second experiment, the level of oxygen in the glovebox atmosphere was allowed to increase to 10%, resulting in complete corrosion of all samples, and highlighting the acceleration of attack by oxygen impurities.

This report presents the results of an additional study of PSOR candidate materials in a simulated reduction vessel environment. The study was designed to confirm the earlier results obtained by Indacochea, et al. [7] and provide additional data at longer test times. The principal objective was to screen several candidate steel alloys (including some not examined in the earlier study) and provide initial corrosion rate information to be used in the vessel design.

Experimental Procedures

Materials

Five ferrous alloys were chosen for evaluation. The alloys needed to possess sufficient high-temperature strength to be used as an ASME pressure vessel at 650°C and also be easily fabricated into a large, cylindrical shape. Three austenitic stainless steels were selected: types 304, 316L, and 347. Two alloy steels were also evaluated: 2.25Cr-1Mo and 9Cr-1Mo-V. The presence of welds (and their associated heat-affected zones) was examined by incorporating a weld into the test capsules. All welds were inert-gas shielded arc welds, with filler metals used as recommended for each material in the AWS code. No post-weld heat treatments were performed; all materials were tested in the as-machined and welded condition.
Corrosion Testing

The corrosion tests were accomplished by filling sealed capsules of each candidate material with a LiCl-Li₂O-Li₃N-Li salt solution and exposing the capsules at 725°C for 30, 60, and 90 days. After exposure, the corrosion of each capsule was assessed via destructive examination. Cylindrical capsules with 0.75 inch outside diameter, 0.063 inch wall thickness, and 3 inch length were machined from each alloy. After machining, each capsule was cut into two halves and welded together, cleaned, and fitted with a 0.75 inch Swagelok cap at the open end. Half of the capsules incorporated a hanging rod from the Swagelok cap with a small piece of porous type 316 stainless steel used in tests with retained Li (see below).

The completed capsules were loaded with the salt constituents LiCl, Li₂O, and Li₃N in a pass-through Ar glovebox, which was maintained at less than 100 ppm oxygen and water vapor. The salt mixture was 95.5 wt% LiCl, 3.5 wt% Li₂O, and 1 wt% Li₃N. Half of the capsules for each material type (those without hanging rods) were loaded with Li metal slightly in excess of that needed to saturate the salt solution. These capsules were referred to as "free Li" capsules since Li metal in excess of the saturation amount was free to float on the surface of the salt. The porous metal pieces attached to the Swagelok end-caps were loaded with Li metal by dipping them into liquid Li and allowing the Li to wick into the pores. These capsules were referred to as "retained Li" since excess Li was held in the porous metal by surface tension and was not available to corrode the capsule material as a free, liquid metal.

After loading with salt and Li, the capsules, with end-caps finger tightened, were de-gassed by heating in a resistance furnace to 725°C for one hour in the Ar glovebox. The Swagelok end-caps were then tightened to seal the capsules. The capsules were re-loaded into the furnace, heated to 725°C, and exposed for a 30 day test interval. An Ar atmosphere containing less than 200 ppm oxygen was maintained during the entire test period.

Post-Test Capsule Examination

At each test interval (30, 60 or 90 days), the furnace was shut off and allowed to cool to room temperature, and one capsule for each combination of material and salt solution was selected for examination. The capsules to be examined were cut into two halves using a pipe cutter. The solidified salt was removed from the bottom halves by draining at 650°C for one hour. A small crust of salt usually remained on the capsules, which was removed by washing in distilled water, followed by a final rinse in ethanol.

Corrosion was assessed primarily by examination of metallographic cross-sections of the capsules using light microscopy. The thickness of the wall was measured using calipers after sectioning. Scanning electron microscopy (SEM) was performed on some sections, coupled with microchemical analysis using energy-dispersive spectroscopy (EDS).

Results

General

At the beginning of the first exposure period, a film of white dust formed on the interior surfaces of the glovebox. The dust was analyzed and shown to contain LiCl, indicating that salt inside one or more capsules was the source. The exposure was halted and the capsules examined and weighed. Some capsules showed signs of salt condensation around the Swagelok seals. The weight changes in the capsules were variable, and were clearly influenced by weight gains due to oxidation (see below). The Swagelok fittings of capsules which showed condensation were tightened, but condensation of salt in the glovebox continued to be observed throughout the exposures.

Despite the relatively low oxygen content in the pass-through Ar glovebox (approximately 100-200 ppm), the exterior surfaces of all capsules were oxidized as a result of the extended high-temperature exposure. The extent of oxidation varied with material type. The 2.25Cr-1Mo and
9Cr-1Mo-V capsules were badly oxidized following exposure. The oxide had spalled in many areas of the capsules. The stainless steel capsules were generally less oxidized than the alloy steel capsules.

Many of the capsules showed signs of severe cold work and subsequent recrystallization on the interior surfaces of the capsules. The cold work was manifest in the austenitic stainless steels as martensite and deformation twins near the inside surface. Very fine grains along the inside surface were a clear result of recrystallization. The cold work damage was exclusive to the stainless steels and apparently resulted from the boring operation used in machining the hollow capsule from rod stock. The microstructural changes due to machining damage made interpretation of corrosion results difficult.

**Type 316L Stainless Steel**

The results for all type 316L stainless steel capsules are summarized in Table 1. The main form of corrosion was intergranular porosity along the inside surface. For some samples the pores were isolated, while in others a continuous network of grain boundary pores was observed. Figure 1 shows an example of connected porosity in Capsule 8 (60 days, free Li). Pores were also observed in the larger-grained weld heat-affected zone (HAZ) and in the weld fusion zone. Porosity in the weld fusion zone favored the ferrite/σ phase. Two of the capsules exposed in the free Li salt solution showed distinct, colored layers along the inside surfaces, as shown in Fig. 2 for Capsule 7 (30 days, free Li). These layers were observed over the entire inside surface, although the thickness in the weld fusion zone was less than in the base metal. SEM examination of these capsules revealed grain boundary porosity extending beyond the distinct layer.

The depth of penetration observed in the base metal was roughly equal for the three capsules exposed to the free Li salt solution. In contrast, the depth of penetration in the capsules exposed to the retained Li salt solution was greater than those exposed to the free Li solution and generally increased with exposure time. Attack in the coarse-grained weld HAZ was severe for all three retained Li capsules, increasing to the entire width of the wall (1500 μm) in the 90 day sample.

The only compositional change resulting from exposure to the salt solution was a depletion of Cr at the very surface of the capsule. In each of the capsules examined in the SEM, a lowering of Cr content to 8-14 wt% from the base metal composition of 17 wt% was observed. The depletion was only found within approximately 5 μm of the surface, and appeared to be more prevalent in areas of higher porosity density. Small crystalline deposits of pure Cr (verified using EDS) were observed along the inside surfaces of several of the 316L capsules (Fig. 2).

**Type 304 Stainless Steel**

The results for type 304 stainless steel capsules are summarized in Table 2. Distinct corrosion layers similar to those observed in the type 316L capsules were present in three type 304 capsules (14, 15, 17). SEM examination of Capsule 14 showed the corrosion layer to be highly porous (Fig. 3). Unlike the type 316L capsules, the porosity did not clearly follow grain boundaries. EDS analysis of this layer showed that it was depleted in Cr. The Cr content in the layer was 2-6 wt%, compared to 20 wt% in the base metal. Corrosion in other type 304 capsules was evident as sporadic porosity visible in unetched cross-sections. The porosity was not clearly aligned along grain boundaries. All type 304 capsules showed signs of severe sensitization resulting from the thermal exposure at 725°C.

The depth of penetration for the type 304 capsules was generally less than for type 316L capsules. In the free Li salt solution, the depth of attack in the base metal increased with time from 25 μm at 30 days to 60 μm at 90 days. Less attack was observed in the weld HAZ and fusion zones, but there was no consistent trend. The depth of penetration in the base metal increased from 30 days to 60 days in the retained Li solution, but no signs of corrosion were
observed in the 90 day capsule. Again, less attack was observed in the weld HAZ and fusion zones.

**Type 347 Stainless Steel**

The results for type 347 stainless steel capsules are summarized in Table 3. Generally less corrosion was observed for this material compared to either types 316L or 304. The form of corrosion typically observed in the type 347 capsules was sporadic porosity observed along the inside wall. Figure 4 shows an example of such porosity in Capsule 19 (30 days, free Li). In some capsules the porosity was clearly aligned along grain boundaries, but in others it appeared to be randomly distributed.

Small reductions in Cr content very near the exposed surfaces were observed in the two capsules examined in the SEM. The Cr content was lowered to 15 wt% from the base metal value of 18 wt% within approximately 5 μm of the surface. No other compositional changes were observed. Pure Cr crystals were deposited along the exposed surfaces of several capsules.

The extent of attack and microstructural alteration for the type 347 capsules was low relative to the other materials tested. The depth of attack in the base metal ranged from 13 to 50 μm. There was no increasing trend of corrosion layer thickness with increasing exposure time, nor were there differences between the free and retained Li salt solutions. The maximum depth of penetration observed was 75 μm, in a weld HAZ (Capsule 23; 60 days, retained Li). In this case the porosity observed was clearly along grain boundaries.

**2.25Cr-1Mo Alloy Steel**

The results for the 2.25Cr-1Mo steel capsules are summarized in Table 4. The outside surfaces were severely oxidized, and three of the capsules had thick oxide layers on the inside surfaces. The presence of oxide, rather than another compound, was confirmed in the SEM using EDS. The salt solution is believed to have been contaminated by oxygen leaking into the capsule through a failed Swagelok seal. Severe oxidation of the exterior surface would disrupt the seal.

The three capsules which did not have thick oxide layers showed little sign of corrosion. Only two capsules showed any sign of attack. The corrosion layer in Capsule 30 (30 days, retained Li) was porous, while corrosion in Capsule 29 (60 days, retained Li) appeared to be intergranular attack rather than porosity formation. The penetration depths observed, 10 μm for Capsule 30 and 30 μm for Capsule 29, were among the lowest for all materials.

**9Cr-1Mo-V Alloy Steel**

The results for the 9Cr-1Mo-V steel capsules are summarized in Table 5. All capsules showed an identical form of corrosion—the decoration of grain boundaries with pores in a region 200-300 μm adjacent to the exposed inner surface (Fig. 5). There was no trend in the depth to which porosity was observed; some capsules exposed for longer times (e.g. Capsule 36) had a smaller depth of attack than capsules exposed for shorter times. The density of porosity appeared to increase with increasing exposure time.

SEM examination of Capsule 32 (60 days, free Li) showed that extensive precipitation occurred in the corrosion zone. The precipitates were acicular, high in Cr and Mo content, and formed both along grain boundaries and in the interior. These precipitates were not observed in the unaffected region of the wall. EDS analysis did not reveal any gross compositional changes due to the corrosion, although the matrix adjacent to the precipitates was depleted in Cr.
Discussion

The results observed have provided a good screening of the candidate materials, but have not enabled the formulation of corrosion rates, since the behavior was erratic. No consistent trends in corrosion extent with time were observed. In particular, several 90 day corrosion capsules showed lower extents than 30 day capsules. It is unclear whether the corrosion process is simply very stochastic, or whether other experimental variables (discussed below) may have influenced the results. Erratic corrosion morphologies were observed in the types 316L, 304, and 347 stainless steels. In some capsules distinct corrosion layers were observed, while other capsules showed no clear layers but rather sporadic porosity.

Two problems with the tests which likely contributed to the erratic behavior are the presence of machining damage on the inside surfaces of some capsules and the presence of significant levels of oxygen in the nominally Ar glovebox during testing. The machining damage resulted from the boring needed to construct a hollow capsule from solid rod stock. Rod stock was used because tube stock was not available in equal sizes for all of the candidate materials. The presence of microstructural changes due to machining damage and recrystallization interfered with identification of changes solely due to corrosion, and hence the measured corrosion extents for these capsules is subject to error. Another issue resulting from the presence of machining damage is that the corrosion rate of the damaged microstructure may be different than that of the baseline structure expected in an actual vessel. There are, however, some cases in which the machining damage clearly did not strongly influence the overall behavior of the capsule. In particular, the extent of corrosion in the 316L and 9Cr-1Mo-V steels was sufficiently large that the effects of machining damage are minimal.

The presence of substantial levels of oxygen in the nominally Ar glovebox environment in which the capsules were exposed also negatively influenced the results of the testing. The oxygen levels during the 30 to 90 day exposure times were approximately 100-200 ppm. The sealed capsules were intended to eliminate glovebox purity as a variable by isolating the salt solution from the glovebox atmosphere. Unfortunately the oxygen levels were sufficient to significantly oxidize the exterior surfaces of all capsules. In the case of the 2.25Cr-1Mo steel, the oxidation was very severe and is believed to have led to gross failure of the Swagelok seals. Other capsules were believed to leak (based on salt condensation in the glovebox), but only in the 2.25Cr-1Mo capsules was oxide identified as a corrosion product. The presence of oxygen invalidated the results from three of the capsules.

The tests were useful in screening the five steels in spite of the problems described above. The results from the type 316L and 9Cr-1Mo-V capsules were not overly influenced by machining damage or oxygen contamination, and both of these materials were badly attacked by the LiCl salt solution. Hence, these two materials are likely not good candidates for use in the reduction vessel. The performance of the type 304 stainless steel was reasonably good in comparison, but the severe sensitization of this material resulting from thermal exposure could be a problem in actual application. The results of these tests do not indicate whether a low-carbon version of 304 would show similarly good corrosion resistance. The poor performance of the type 316L capsules compared to the type 304 capsules may have been related to the difference in carbon content, but this is unclear.

Both the type 347 stainless steel and the 2.25Cr-1Mo alloy steel may be good candidate materials worthy of further investigation. Both showed little or no sign of corrosion, with the exception of the 2.25Cr-1Mo capsules that apparently leaked. The 2.25Cr-1Mo capsules that were not badly oxidized showed very little corrosion. The type 347 capsules also showed relatively little corrosion, although the results for this material were somewhat erratic and may have been influenced by the presence of machining damage in some capsules. The inherent stability of type 347 stainless steel with respect to sensitization at the operating temperature of the reduction vessel is a strong point.

Due to the problems with some of the capsules and the generally erratic nature of the results obtained, it was not possible to formulate corrosion rates for the different materials in the LiCl salt solution. The mechanism of corrosion for all five materials appears to be depletion of Cr from the alloy, preferentially at grain boundaries. Depleted levels of Cr near the surface were
observed in all of the stainless steel alloys, accompanied by the formation of pores along grain boundaries. Grain boundary porosity was also observed in the 9Cr-1Mo-V alloy, although Cr depletion was not clearly measured. Additional support for the dissolution of Cr into the salt solution is provided by the precipitation of Cr crystals along the inside surfaces of some capsules.

Depletion of Cr from iron-nickel-chromium alloys is identified as a mechanism of corrosion in high-temperature molten salt systems in ref. [5]. Cr dissolves into the salt solution, leaving a Cr-depleted layer at the surface. Vacancies resulting from depletion and diffusion eventually form voids, usually at grain boundaries. This mechanism is identical to that observed in the present tests. One reason for the better performance of the 2.25Cr-1Mo alloy is that the initial Cr amount is lower, resulting in smaller gradients to drive diffusion and vacancy formation when Cr is depleted from the surface. An increase in Cr content from 10 to 30% was reported to result in a seven-fold increase in corrosion rate [5], a trend which is consistent with the results of this study.

The behavior in these tests is contrary to that observed in similar studies by Indacochea, et al. [7]. They tested types 316L, 422, and 430 stainless steels, 2.25Cr-1Mo alloy steel, and pure Ta at 725°C in a LiCl salt solution with 3.5 wt% Li$_2$O and 1 wt% Li$_3$N saturated with Li metal. Contrary to the results of the current study, no evidence of corrosion was found in the steel alloys after 30 days. Differences in the test configuration and sample characteristics may have contributed to the different behavior observed. In ref. [7], the test coupons were flat plates machined from sheet stock and were partially immersed in the salt solution, which was contained in a magnesia crucible. The crucible was located in a purified Ar glovebox, and Ar gas was continually bubbled through the salt solution during the test. This is in contrast to the use of capsules machined from bar stock that were placed in relatively impure Ar environment in the present study. The machining may have increased the susceptibility of the alloys to corrosion, and oxygen in the glovebox environment may have contaminated the salt solution, also giving rise to increased corrosion. Oxygen contamination was likely a factor in several of the 2.25Cr-1Mo capsules, however, oxygen was not observed in any of the other capsules that were examined in the SEM. It is therefore unclear whether oxygen contamination was a factor in all tests.

Conclusions

The results of this series of corrosion tests on five steels that are candidates for use in the Li oxide reduction vessel may be summarized in the following points:

1. All steels showed some sign of corrosion or interaction with the molten salt solution. The primary form of corrosion observed was the depletion of Cr from the exposed surface accompanied by formation of grain boundary porosity, consistent with previous observations of steel corrosion in molten salt systems.
2. The type 316L stainless steel and 9Cr-1Mo-V alloy steel showed the greatest corrosion extent, in one capsule nearly resulting in full penetration of the capsule wall.
3. Considerably less corrosion was observed for the type 304 stainless steel, however, the microstructure of this steel was severely sensitized by the thermal exposure.
4. Little corrosion was observed for the type 347 stainless steel and the 2.25Cr-1Mo alloy steel. These two alloys are the best candidates for further evaluation.
5. The extents of corrosion observed were too erratic to formulate meaningful corrosion rates.

Acknowledgements

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References


### Table I. Type 316L Stainless Steel Capsules (Type 316L Weld Filler)

<table>
<thead>
<tr>
<th>Capsule ID</th>
<th>Exposure Time (d)</th>
<th>Li Form</th>
<th>Base metal (µm)</th>
<th>Weld fusion (µm)</th>
<th>Weld HAZ (µm)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>30</td>
<td>Free</td>
<td>100</td>
<td>30</td>
<td>105</td>
<td>Grain boundary porosity. Cr crystals observed on inside surface. Cr depleted to 8-14 wt% within ~5 µm of exposed surface.</td>
</tr>
<tr>
<td>8</td>
<td>60</td>
<td>Free</td>
<td>90</td>
<td>225</td>
<td>90</td>
<td>Grain boundary porosity. Cr depleted to 8-12 wt% within ~5 µm of exposed surface.</td>
</tr>
<tr>
<td>9</td>
<td>90</td>
<td>Free</td>
<td>90-95</td>
<td>45</td>
<td>235</td>
<td>Distinct 90 µm layer. Deeper grain boundary porosity in weld HAZ.</td>
</tr>
<tr>
<td>10</td>
<td>30</td>
<td>Retained</td>
<td>100</td>
<td>420</td>
<td>400</td>
<td>Grain boundary porosity. Deformation and recrystallization. Cr crystals on inside surface.</td>
</tr>
<tr>
<td>11</td>
<td>60</td>
<td>Retained</td>
<td>175</td>
<td>none</td>
<td>450</td>
<td>Grain boundary porosity.</td>
</tr>
<tr>
<td>12</td>
<td>90</td>
<td>Retained</td>
<td>480</td>
<td>1050</td>
<td>1500</td>
<td>Grain boundary porosity across entire section in weld HAZ.</td>
</tr>
</tbody>
</table>

### Table II. Type 304 Capsules (Type 308 Weld Filler)

<table>
<thead>
<tr>
<th>Capsule ID</th>
<th>Exposure Time (d)</th>
<th>Li Form</th>
<th>Base metal (µm)</th>
<th>Weld fusion (µm)</th>
<th>Weld HAZ (µm)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>14</td>
<td>60</td>
<td>Free</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>Distinct, highly porous layer. Cr reduced to 2-6 wt% in layer.</td>
</tr>
<tr>
<td>15</td>
<td>90</td>
<td>Free</td>
<td>60</td>
<td>none</td>
<td>20</td>
<td>Distinct corrosion layer. thinner in weld HAZ.</td>
</tr>
<tr>
<td>16</td>
<td>30</td>
<td>Retained</td>
<td>35</td>
<td>10</td>
<td>20</td>
<td>Deformation and recrystallization.</td>
</tr>
<tr>
<td>17</td>
<td>60</td>
<td>Retained</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>Distinct corrosion layer.</td>
</tr>
<tr>
<td>18</td>
<td>90</td>
<td>Retained</td>
<td>none</td>
<td>none</td>
<td>none</td>
<td>No sign of corrosion.</td>
</tr>
</tbody>
</table>
### Table III: Type 347 Stainless Steel Capsules (Type 347 Weld Filler)

<table>
<thead>
<tr>
<th>Capsule ID</th>
<th>Exposure Time (d)</th>
<th>Li Form</th>
<th>Corrosion Extent (µm)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>19</td>
<td>30</td>
<td>Free</td>
<td>Base metal: 45</td>
<td>Sporadic porosity near capsule bottom. Reduction in Cr content to 15 wt% in porous region. Cr crystals on inside surface.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld fusion: none</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld HAZ: none</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>60</td>
<td>Free</td>
<td>Base metal: 30</td>
<td>Layer of porosity along inside surface.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld fusion: 30</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld HAZ: 30</td>
<td></td>
</tr>
<tr>
<td>21</td>
<td>90</td>
<td>Free</td>
<td>Base metal: 50</td>
<td>Distinct layer of porosity along inside surface. Grain boundary porosity in weld HAZ. Cr crystals on inside surface.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld fusion: none</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld HAZ: 50</td>
<td></td>
</tr>
<tr>
<td>22</td>
<td>30</td>
<td>Retained</td>
<td>Base metal: 20</td>
<td>Sporadic porosity along inside surface. Deformation and recrystallization.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld fusion: none</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld HAZ: 10</td>
<td></td>
</tr>
<tr>
<td>23</td>
<td>60</td>
<td>Retained</td>
<td>Base metal: 40</td>
<td>Sporadic grain boundary porosity. Deformation and recrystallization. Cr reduced to 15 wt% within 5 µm of exposed surface. Cr crystals on inside surface.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld fusion: none</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld HAZ: 75</td>
<td></td>
</tr>
<tr>
<td>24</td>
<td>90</td>
<td>Retained</td>
<td>Base metal: 13</td>
<td>Very sporadic porosity; maximum depth 13 µm.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld fusion: none</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Weld HAZ: none</td>
<td></td>
</tr>
</tbody>
</table>

### Table IV: 2.25Cr-1Mo Steel Capsules (ER90S-B3 Weld Filler)

<table>
<thead>
<tr>
<th>Capsule ID</th>
<th>Exposure Time (d)</th>
<th>Li Form</th>
<th>Corrosion Extent (µm)</th>
<th>Notes</th>
</tr>
</thead>
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<td>25</td>
<td>30</td>
<td>Free</td>
<td>Base metal: none</td>
<td>No corrosion.</td>
</tr>
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<td></td>
<td></td>
<td>Weld fusion: none</td>
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<td></td>
<td>Weld HAZ: none</td>
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</tr>
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<td>26</td>
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<td>Base metal: 70</td>
<td>Thick oxide layer: leaking capsule.</td>
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<td>Weld HAZ: 70</td>
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<td>90</td>
<td>Free</td>
<td>Base metal: 150</td>
<td>Thick oxide layer: leaking capsule.</td>
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<td>Weld fusion: 150</td>
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<td>Weld HAZ: 150</td>
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<td>Retained</td>
<td>Base metal: 10</td>
<td>Sporadic porosity.</td>
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<td>Weld fusion: none</td>
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<td></td>
<td></td>
<td>Weld HAZ: none</td>
<td></td>
</tr>
<tr>
<td>29</td>
<td>60</td>
<td>Retained</td>
<td>Base metal: 30</td>
<td>Grain boundary penetration.</td>
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<td>Weld fusion: none</td>
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<td>Weld HAZ: 30</td>
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<td>Weld fusion: 230</td>
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<td>Weld HAZ: 230</td>
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Table V. 9Cr-1Mo-V Steel Capsules (ER90S-B9 Weld Filler)

<table>
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<tr>
<th>Capsule ID</th>
<th>Exposure Time (d)</th>
<th>Li Form</th>
<th>Corrosion Extent (μm)</th>
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<td>Free</td>
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<td>Free</td>
<td>Base metal: 300</td>
<td>Grain boundary porosity. Acicular precipitates in affected region. No Cr depletion.</td>
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<td>Weld HAZ: 300</td>
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<td>Grain boundary porosity.</td>
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<td>Retained</td>
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<td>Weld HAZ: 225</td>
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<td>Weld fusion: 200</td>
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<tr>
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<td>90</td>
<td>Retained</td>
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<td>Grain boundary porosity.</td>
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<td>Weld fusion: 130</td>
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<td>Weld HAZ: 130</td>
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</table>

Figure 1: Connected grain boundary porosity in type 316L stainless steel capsule (Capsule 8; 60 days, free Li). Exposed surface is at bottom.
Figure 2: Distinct corrosion layer observed in type 316L Capsule 7 (30 days, free Li). Also shown is a triangular-shaped Cr crystal deposited on the exposed surface (top).

Figure 3: SEM image of distinct corrosion layer in type 304 Capsule 14 (60 days, free Li). Exposed surface is at bottom.
Figure 5: Sporadic porosity observed in type 347 Capsule 19 (30 days, free Li). Exposed surface is at top.

Figure 6: Grain boundary porosity observed in 9Cr-1Mo-V Capsule 33 (90 days, free Li). Exposed surface is at top.