

**PbSCC OF ALLOY 800NG STEAM GENERATOR TUBING IN ALKALINE ENVIRONMENTS**

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**ABSTRACT**

Laboratory testing suggest that Alloy 800NG steam generator tubing is susceptible to lead stress corrosion cracking (PbSCC) when exposed to lead contaminated high pH conditions. Assessing the range of pH values where Alloy 800NG is susceptible to PbSCC is important to help understand the range of operating values that need to be maintained to prevent PbSCC from occurring in operating steam generators. In this program, two pH values were used and the PbSCC resistance of Alloy 800NG was measured for Alloy 800NG in the solution annealed (Alloy 800NG) and cold worked (CW Alloy 800NG) conditions in lead contaminated NaCl solutions of pH\(_{330°C}\) 8.5 and 9.5 meant to simulate realistic contaminated crevice conditions. Results showed that Alloy 800NG was susceptible to cracking at both pH values, but that at pH\(_{330°C}\) 8.5 there was a significant incubation time before the onset of cracking. The results will be used to improve guidance on operation of steam generators utilizing Alloy 800NG tubing.

**Keywords:** Stress Corrosion Cracking, ODSCC, PbSCC, Lead, Steam Generator, Alloy 800, Caustic, Chloride, Secondary Water Chemistry

**1.0 INTRODUCTION**

Intergranular attack/stress corrosion cracking (IGA/SCC) has been observed in Pressurized Water Reactor (PWR) Alloy 600 steam generator tubes since the early 1970’s [1]. Examinations of pulled tubes have found cracking in the upper bundle, free span superheated regions of once through steam generators (OTSGs) and in the tube/tube support plate intersections and at the top of tubesheet crevices of recirculating steam generators (RSGs). These examinations suggested that cracking occurs in flow restricted regions and is caused by the concentration of impurities from the secondary water. Deposit buildup in the tube/tube support plate crevices is the cause of flow restriction and chemical concentration in RSGs. In OTSGs, concentration of impurities may occur in the porous deposits on the freespan where liquid droplets, transported in the slightly superheated steam, can deposit and form a concentrated liquid phase. Hideout return data and a comparison of chemical analyses of feedwater and steam condensate suggested that the concentrated liquid phase is frequently alkaline containing species such as Na, Al, Si, Ca, K, and Mg [2]. Also, surface analyses of the fracture faces of cracks in many pulled tubes have detected Cr/Ni ratios consistent with an alkaline environment [3] [4]. Thus, since the earliest observations, most IGA/SCC observed in steam generator tubes was attributed to caustic cracking, or caustic cracking further accelerated by a second species.

The morphology of the attack observed in pulled tubes resembled that observed in the laboratory produced by caustic solutions in early Alloy 600 test programs. There were large areas of the outside diameter of the tubing where all or most grain boundaries were penetrated are like the IGA produced in the laboratory at open circuit potential (OCP) and at potentials slightly more oxidizing than the OCP. The more isolated deep intergranular penetrations resemble the caustic SCC produced at oxidizing conditions (high electrochemical potentials) [5] [6].

The present strategy for mitigating IGA/SCC is based on the premise that crack initiation and propagation rates depend on pH and the electrochemical potential. Accordingly, plants have increased water purity (As Low As Reasonably Achievable, ALARA chemistry) and reduced oxygen ingress. Plants have also
adopted the practice of injecting sufficient hydrazine to maintain reducing conditions (low electrochemical potentials). Plants maintain secondary system and steam generator bulk water pH by the addition of amines to the feedwater. Some plants have also attempted to control crevice pH to near neutral, by molar ratio control; i.e., controlling the ratio of sodium to chloride and sulfate in the bulk water to prevent highly acidic or caustic conditions from developing [7]. These measures have been successful in reducing the rate of degradation in Alloy 600 tubes affected by IGA/SCC in some plants. However, other plants using the same measures have continued to experience IGA/SCC. The reason for this variability is not well understood, but there is mounting evidence that lead (Pb) is the cause [8] [9] [10].

Pulled tube examinations performed over the years have implied that Pb may have been involved in numerous occurrences of IGA/SCC, but definitive evidence was generally lacking. High magnification analytical transmission electron microscopy (ATEM) has been used to examine degraded tube specimens and has now revealed Pb within porous, nanocrystalline, degraded grain boundary oxides that were several nanometers wide [4]. The localized appearance of Pb means that Pb may have been involved in more cases of SCC than previously believed where spatial resolution of detection techniques was insufficient to resolve it. SCC due to the presence of Pb is termed PbSCC. Prior testing [11] has evaluated PbSCC at pH330°C of 5, 7. And 9.1 and has focused on Alloy 600MA, Alloy 600TT and Alloy 690TT.

Alloy 800 was developed in the 1950s. At the time, nickel was designated as a strategic material, subject to disruptions in availability. In response to this, Alloy 800 was intended to fill the need for a corrosion resistant alloy that can be used at high temperatures with relatively low nickel content (for example, less than the ~75% Ni content of Alloy 600). It has good strength and excellent resistance to corrosion in aqueous environments at elevated temperature [12]. A modified version of Alloy 800 (Alloy 800NG) has been used as the steam generator tubing material in numerous plants around the world. Additionally, some Alloy 800NG also includes a 4% cold draw step to increase mechanical properties and shot peening using glass beads on the OD to introduce further compressive stresses and increase SCC resistance. Overall, Alloy 800NG has performed well in steam generators.

Based on the papers reviewed [8] [13] [14], Alloy 800NG appears to be susceptible to PbSCC but the exact conditions were it is susceptible have not been fully determined. At neutral or near-neutral conditions, Alloy 800NG is highly resistant to PbSCC, however at both acidic and alkaline conditions Alloy 800NG has been shown to be susceptible to PbSCC. There has been limited testing to define the pH where Alloy 800NG first starts to become susceptible to PbSCC. The latter region is more of interest since alkaline environments are more likely in steam generators. The purpose of this testing is to investigate the effect of alkaline pH values on PbSCC resistance, to investigate the effect of applied potential on PbSCC resistance, and to investigate the surface films on Alloy 800NG to help elucidate the cracking mechanism.

2.0 EXPERIMENTAL SETUP

Testing was performed using highly strained reverse U-bend (RUB) specimens fabricated from Alloy 800NG. The test environments were relatively simple NaCl-based solutions with 500 ppm Pb with NaOH additions to achieve different pH values at 330°C.

2.1 Test Materials

The Alloy 800NG specimens tested were from two heats, one heat in the solution annealed condition (Alloy 800NG) and one heat with 4% cold drawing and shot peening (CW Alloy 800NG). The outside diameter (OD) of supplied Alloy 800NG tubing is 15.88 mm (5/8 inch) with a wall thickness of 1.145 mm (0.0447 inch), while the OD of supplied CW Alloy 800NG tubing is 19.05 mm (3/4 inch) with a wall thickness of 1.143 mm (0.0450 inch). The vendor supplied the composition of the Alloy 800NG material, with the exception of the Pb. Luvak Inc. analyzed the Alloy 800NG material for Pb and performed a chemical analysis of CW Alloy 800NG material. Table 1 shows the chemical compositions of the two
heats. Table 2 shows the vendor supplied mechanical properties for the Alloy 800NG material and the room temperature mechanical properties measured for CW Alloy 800NG measured by Westmoreland Mechanical & Research, Inc.

### 2.2 Test Environments

The test solutions for this program were developed from analysis of hideout return data and reviews of plant operating data. These data indicate that the concentrated liquid phase in support plate crevices and tubesheet crevices in recirculation steam generators (RSG) has a complex chemistry composed of many species. However, data suggest that the main solute is sodium chloride. Sodium and chloride are the primary species found in RSG blowdown during power operation and frequently are the highest concentration species seen in hideout return (HOR) evaluations. Although species like silica, sulfate, calcium, aluminum, etc., are also seen in HOR, results suggest that most of this return is from adsorption release and/or deposit dissolution rather than release from concentrated liquid solutions in flow restricted regions.

The test solutions selected for this program are a 3m NaCl base solution with the pH adjusted with NaOH and 500 ppm Pb added as PbO to result in pH values calculated to be 8.5 and 9.5 at 330°C as shown in Table 3. Thermodynamic considerations suggest that this NaCl concentration is reasonable for that in the porous deposits in tube/TSP crevices of RSGs. The simple chloride solution was chosen so that the high temperature pH of the solution could be calculated as accurately as possible. Table 3 also shows the distribution of Pb species in each environment as computed by MULTEQ [15]. Due to previous use of the available autoclaves with Pb contaminated solution testing, it was not practical to perform testing in an environment where Pb contamination was reduced to a sufficiently low level to be considered a Pb free environment.

### 2.3 Test Procedures

Tests were performed using Reverse U-Bend (RUB) specimens. These were fabricated by splitting 12.7 cm (5 inch) long segments of the tube longitudinally using the fixture shown in Figure 1a to ensure the cut was made along the axis of the tubing. The split tube sections were bent around a “donut” shaped mandrel shown in Figure 1b. Separate mandrels were used for the two types of Alloy 800NG material. They were designed to produce the same strain at the apex of the RUB specimens as that in the Alloy 600MA, Alloy 600TT, and Alloy 690TT RUB specimens used in a previous program [16]. The bend radius was 1.49 cm (0.587 inches) for the Alloy 800NG tubing and 1.50 cm (0.591 inches) for the CW Alloy 800NG tubing where the differences in mandrel size account for differences in tubing dimensions and mechanical properties. When the specimen is removed from the mandrel, the legs are allowed to relax. The legs are then re-strained by inserting an Alloy 600 bolt through drilled holes at the base of the legs and tightening the bolt until the legs are parallel. This produces a RUB specimen with the original inside surface of the tube in tension.

Specimens were electrically isolated and mounted in a titanium autoclave (shown in Figure 2) with the test solution. The system was deaerated using a 95% Argon/5% Hydrogen cover gas with at least three cycles of pressurizing (to between 1.4 to 13.6 MPa (200 to 2000 psia)), holding for 30 minutes, and depressurizing. Before starting the test, the autoclave was pressurized to 1.4 MPa (200 psia) at room temperature with the same cover gas, sealed, and heated to the test temperature of 330°C. The hydrogen gas in the cover gas results in a final dissolved hydrogen concentration of 6 ppm. This value was chosen for consistency with previous test programs [16]. While the dissolved hydrogen on the secondary side is generally low, the hydrogen centration inside tight cracks of tubing expected to be significant due to diffusion from the primary coolant. Specimens were periodically removed from the autoclave for inspection and the autoclave reloaded. The solution in the autoclave was refreshed for each restart.
2.4 Post Test Examinations
After exposure, samples were destructively examined using metallography examination of mounting, sectioning, and polishing to determine the maximum crack length. Note that the full thickness of the specimens is approximately 1140 µm and this represents a through wall crack.

Specimens for surface analysis were one half inch sections cut from segments of tubes that had been split longitudinally. Films for analysis were produced by exposing the specimens to the PbSCC test conditions for 48 hours. After the specimens were removed from the autoclaves, they were rinsed in demineralized water, dried and placed in the spectrometer. Auger analysis was performed using a Perkin Elmer model 600 Auger spectrometer. A 500 V electron beam was used. Spectra were taken at 1 eV per step with 100 mSec per step. A differentially pumped ion gun was used for in-depth profiling. The sputtering rate was 50Å per minute, which was determined from measurements on SiO₂ films on known thickness. Spectra were analyzed with RBD AugerScan software.

3.0 RESULTS

3.1 Open Circuit Potential, pH₃₃₀°C 9.5
Maximum crack depth as a function of exposure time for Alloy 800NG specimens exposed to Pb containing solutions at pH₃₃₀°C of 9.5 at the OCP are shown in Figure 3. The maximum exposure time was 1112 hours where through-wall cracking was observed. For the CW Alloy 800NG, through thickness cracking was observed after only 432 hours of exposure where four specimens had all cracked through-wall as shown in Figure 4. For Alloy 800NG, intermediate time tests of 200 hours show partial crack growth indicating that through-wall cracking was rapid, but did take some time grow.

Cross section examination of the cracks that formed after exposure at pH₃₃₀°C of 9.5 show some distinctive features of the cracks that form and are shown in Figure 5. First, most of the deeper cracks have an oxide hemispherical indentation on the surface. This indentation into the surface of the specimen can be seen in Figure 5a. The indention is filled with an oxide and the oxide has the appearance of ghost cracks as well. The second distinctive feature is that near the surface of the specimens, the cracking appears to be primarily transgranular. The third distinctive feature is that as cracks penetrate deeper into the specimens, the cracks appear to become more branched and intergranular as can be seen in Figure 5b.

3.2 Open Circuit Potential, pH₃₃₀°C 8.5
Maximum crack depth as a function of exposure time for Alloy 800NG specimens exposed to Pb containing solutions at pH₃₃₀°C of 8.5 at the OCP are shown in Figure 6. Under OCP, cracks appear and grow in relatively short times. For the three applied potentials (-50 mV, +50 mV, and
+75 mV), cracks also appear and grow in short time. The crack growth rates appear to be approximately the same or slightly faster with the applied potentials.

### 3.4 Auger Results

The chemical composition of the surface oxides for Alloy 800NG samples were measured as a function of sputtering depth after exposures for both pH\(_{330\,^\circ\text{C}}\) 9.5 (Figure 10) and 8.5 (Figure 11). For exposure at pH\(_{330\,^\circ\text{C}}\) of 9.5, a maximum sputter depth of approximately 7500 Å was used. The film formed on the surface of Alloy 800NG at this depth still contained significant amounts of oxygen, Pb, and Ti. Ti is believed to come from the autoclave body itself. The concentration of Pb and Ti through the observed surface oxide thickness suggests that there were incorporated during growth of the oxide film or that the film is very porous letting the solution extend toward the surface. There is a slight increase in chromium at approximately 7000 Å indicating that some oxide formed at that depth could be richer in Cr than in other parts of the oxide surface.

For exposures at pH\(_{330\,^\circ\text{C}}\) of 8.5, a maximum sputter depth of approximately 6000 Å was used. At this depth, the oxygen concentration is decreasing rapidly indicating the end of the oxide. Also, Pb and Ti, which were present throughout the entire oxide at pH\(_{330\,^\circ\text{C}}\) 9.5, were only present in the first ~1000 Å of the oxide film at pH\(_{330\,^\circ\text{C}}\) 8.5. Based on the rapid decrease of Pb and Ti in the top surface regions, it is speculated that they deposited on the surface during cooling and were not incorporated into the oxide as at the higher pH.

### 4.0 DISCUSSION

The results of the testing on Alloy 800NG can be compared to other tubing alloys from a prior test program [16]. For testing at pH\(_{330\,^\circ\text{C}}\) of 9.5, Figure 12 compares the maximum crack depths as a function of time for Alloy 800NG, CW Alloy 800NG, Alloy 690TT, Alloy 600MA, and Alloy 600TT all at OCP. From this figure, the crack growth rate of Alloy 800NG is higher than the three nickel base alloys. This suggests that in a Pb contaminated environment at high pH values, Alloy 800NG should be less resistant than other common steam generator tubing environments. Utility experience with Alloy 800NG has not shown widespread cracking. Therefore, either the contamination levels of Pb in these steam generators have not reach sufficient levels or the pH in crevice locations have not reached high enough values in operating plants to produce this type of behavior.

For testing at pH\(_{330\,^\circ\text{C}}\) of 8.5, Figure 13 compares the maximum crack depths as a function of time for the same alloys all at OCP. For the three nickel base alloys, Alloy 690TT shows complete resistance to cracking and no evidence of cracking was observed at times exceeding 5000 hours. For Alloy 600MA and Alloy 600TT, the cracking increases at a linear rate and appears to start at near zero time. The behavior of Alloy 800NG and CW Alloy 800NG, where a 3000 hour incubation was observed is significantly different. Further, the time between no crack growth and through-wall cracking for Alloy 800NG and CW Alloy 800NG is as short as a few hundred hours, similar to growth rates in pH\(_{330\,^\circ\text{C}}\) 9.5 solutions. There is no clear explanation for the observed incubation time. Since autoclaves were occasionally and randomly refreshed, it does not appear to be purely an experimental artifact. Possibilities include an aging effect in the oxide film which decreases its ductility or the time needed for some concentrating effect at the surface-oxide interface to build Pb to sufficient levels for cracking to occur. Further experimentation will be needed to understand this phenomenon.

The crack growth rate of CW Alloy 800NG is higher than non-cold worked Alloy 800NG at both tested pH values. This suggests that this material is less resistant to PbSCC. However, note that the stress state of the RUB specimens is considerable different than the as-manufactured tubing. The surface stresses are especially different and the goal of the shot peening is to put the outer surface in compression, rather than the tension surface state that the RUB specimen type imposes. However, these tests do suggest that the additional cold work from the CW Alloy 800NG is not always beneficial to reducing crack growth when the stress state is disturbed from the as-manufactured state.
The applied potential testing indicates that cracking behavior may be accelerated for potentials more oxidizing than the OCP since the highest crack growth rate corresponds to the testing at +75 mV. The test with a more reducing potential is not significantly different than the behavior at OCP. Since the crack growth rate appears to be accelerated as the potential becomes more oxidizing, this supports the technical bases for operating and maintaining steam generators under reducing conditions.

The oxide formed on the surface of Alloy 800NG is different between the two tested pH values. For the pH$_{330°C}$ 9.5, the surface had indentations filled with oxide products and the Auger analysis showed that Pb was incorporated through the thickness of the surface oxide. The Cr/Ni ratio (shown in Figure 14a) at pH$_{330°C}$ 9.5 shows that the Cr/Ni ratio is above the alloy metal ratio and stays high for entire measured thickness. A low Cr/Ni ratio is expected for high pH values where Cr is expected to selectively dissolve into the solution. The Pb appears to alter the stability of the Cr containing surface oxide resulting in an oxide film which is not protective of the underlying surface.

For pH$_{330°C}$ 8.5, the oxide appeared thinner and Pb wasn’t incorporated through the entire thickness. The Cr/Ni ratio (shown in Figure 14b) starts low where the Pb was observed in the oxide film, increases, and then decreases to approximately the alloy metal value. This indicates that the end of the Auger analysis for this pH is basically back to bare metal and is thinner and that the oxide. The oxide formed on the metal at pH$_{330°C}$ 8.5 appears significantly more protective.

CONCLUSIONS

The SCC behavior of Alloy 800NG, and cold worked Alloy 800NG (CW Alloy 800NG) were assessed in alkaline NaCl solutions with lead (Pb). Assessing the behavior of these alloys on the secondary side of the steam generator is particularly important since the factors influencing SCC can be more aggressive than on the primary side of a PWR (e.g., in-service deformation, cold work during installation, pH, contaminants, and electrochemical potential). The significant conclusions from this test program are:

1. Alloy 800NG is susceptible to PbSCC in alkaline environments. At pH$_{330°C}$ 9.5, the observed crack growth rates were higher than for Alloy 600MA, Alloy 600TT, and Alloy 690TT.
2. There was a significant incubation time before the onset of cracking in Alloy 800NG at pH$_{330°C}$ 8.5, but the actual crack growth rate after the onset of cracking was significant.
3. CW Alloy 800NG may be more susceptible to cracking than non-cold worked Alloy 800NG, especially if the intended surface compressive stress state of the alloy is disturbed.
4. The effect of applied potential on Alloy 800NG supports operating steam generators under reducing conditions.
5. The surface oxide film changes with pH and the changes correspond with differences in PbSCC behavior. The oxide film formed at pH$_{330°C}$ 9.5 appears significantly less protective than the film formed at pH$_{330°C}$ 8.5.

REFERENCES


Table 1: Chemical composition (in weight percent) for heats used in this study

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<tr>
<th>Alloy</th>
<th>C</th>
<th>Mn</th>
<th>S</th>
<th>P</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Cu</th>
<th>Co</th>
<th>Al</th>
<th>Ti</th>
<th>Fe</th>
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<td>21.87</td>
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Table 2: Room temperature mechanical properties and grain size for heats used in this study

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<th>Tensile Strength MPa (ksi)</th>
<th>Hardness (HRB)</th>
<th>Elongation (%)</th>
<th>Grain Size</th>
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<td>620 (90)</td>
<td>78</td>
<td>41</td>
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<tr>
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<td>677 (98.3)</td>
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Table 3: Test solutions used in this study

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Figure 1: Fixtures used to (a) split tubing and (b) bend tubing to produce reverse U-bend (RUB) specimen with example RUB specimen

Figure 2: Titanium autoclave and RUB sample holder lid
Figure 3: Maximum crack depth after exposures to Pb contaminated solution at 330°C and pH_{330°C} of 9.5 at OCP

Figure 4: Photograph of CW Alloy 800NG after exposure to the pH_{330°C} 9.5 at OCP for 432 hours showing through thickness cracking
Figure 5: Micrographs of Alloy 800NG showing PbSCC after exposure to the pH$_{330^\circ C}$ 9.5 at OCP for 912 hours showing (a) oxide filled indentations at crack initiation sites and transgranular cracks near the surface and (b) intergranular cracks for deep cracks.

Figure 6: Maximum crack depth after exposures to Pb contaminated solution at 330°C and pH$_{330^\circ C}$ of 8.5 at OCP. Note that lines are not fit lines and are for reference only.
Figure 7: Micrographs of CW Alloy 800NG showing absence of PbSCC after exposure to the pH$_{330^\circ C}$ 8.5 at OCP for 3120 hours with (a) full thickness and (b) near surface region.

Figure 8: Micrographs of CW Alloy 800NG showing PbSCC after exposure to the pH$_{330^\circ C}$ 8.5 at OCP for 3596 hours with (a) full thickness and (b) transgranular crack tip region.
Figure 9: Maximum crack depth after exposures to Pb contaminated solution at 330°C and pH_{330°C} of 9.5 as a function of applied potential

Figure 10: Auger composition as a function of depth for Alloy 800NG after exposure to Pb Contaminated solution at 330°C and pH_{330°C} of 9.5
Figure 11: Auger composition as a function of depth for Alloy 800NG after exposure to Pb contaminated solution at 330°C and pH of 8.5.

Figure 12: Comparison of maximum crack depth for Alloy 800NG, CW Alloy 800, Alloy 690TT, Alloy 600MA, and Alloy 600TT at pH330°C of 9.5 all at OCP.
Figure 13: Comparison of maximum crack depth for Alloy 800NG, CW Alloy 800, Alloy 690TT, Alloy 600MA, and Alloy 600TT at pH_{330°C} of 8.5 all at OCP.

Figure 14: Cr to Ni ratio after exposure for 48 hours at pH_{330°C} (a) 9.5 and (b) 8.5.