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Evaluation of SCC Test Methods for
Inconel 600 in Low Temperature
Aqueous Solutions

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Abstract

In late 1981, widespread leakage was encountered in Alloy 600 steam generator tubing at the Three Mile Island Unit 1 nuclear power plant. The phenomenon was identified as low temperature intergranular stress-corrosion cracking (SCC) initiated from the inner surfaces of the tubes exposed to the primary coolant. A testing program was initiated to examine the material and environmental factors relevant to these failures, which were found to be associated with sensitization of the material and contamination of the coolant by air and sodium thiosulfate. The test solutions contained 1.3% boric acid with various additions of sulfur compounds and lithium hydroxide. Constant extension rate testing was used as the primary tool to examine environmental effects such as the inhibition of cracking by lithium hydroxide. Important effects of crack initiation frequency on the specimen potential (and therefore crack velocity) are demonstrated. In some of these tests, straining was interrupted following crack initiation and the effects of further environmental additions monitored by following the load decay at constant total strain. This procedure generally resulted in a different ranking of the environments. U-bend tests showed that the ranking of different sulfur-containing environments was also highly dependent on the metal heat treatment.

Introduction

Stress-corrosion cracking of Alloy 600 at temperatures below 100°C was largely a laboratory phenomenon until 1981, when multiple tube penetration by circumferential cracks occurred in the steam generators of a non-operating pressurized water reactor [1]. Several thousand cracks had grown to at least 0.5 mm depth, of which a high proportion had arrested after progressing 80-90% through the tube walls. Contamination of the borated primary coolant by sodium thiosulfate seems to have been the principal environmental factor involved. In addition, sulfur-rich deposits in the tubes may indicate an important role of earlier thiosulfate intrusion during high temperature operation, and could themselves give rise to metastable dissolved species by reaction with air and water. The chemical state of the sulfur in the aqueous phase at the time of cracking is not known with certainty; however it may be safely assumed that a metastable form such as thiosulfate or tetrathionate was present. The aqueous concentration of sulfur was probably around 0.7 ppm, equivalent to $1.1 \times 10^{-5} \text{M Na}_2\text{S}_2\text{O}_3$. The tubing was sensitized by its stress relief treatment following fabrication of the steam generators, and was subject to longitudinal stresses. During temperature excursions in the period preceding obvious tube damage, these stresses may have varied in a manner conducive to SCC initiation. However, knowledge of the time at which air first intruded is also critical. In this investigation we have used several test techniques to examine the sulfur-induced SCC of sensitized Inconel 600, at temperatures from 22 to 95°C. Details of the influence of environmental and metallurgical variables are given in two further papers.[2,3]

Testing philosophy

In this work the constant extension rate (CER) test was used to rank various dilute environments as rapidly as possible, using a constant metallurgical condition. In order to distinguish between genuine environmental effects and artifacts peculiar to the CER test, an interrupted

test was also used. Straining was halted following obvious crack initiation, and additions were made to the environment while following the load relaxation due to crack propagation. In this fashion it was possible to establish the environmental alterations necessary to arrest a constant number of propagating cracks at moderate stress intensities. These critical inhibitive solution compositions are conservative in the sense that, if a propagating crack is arrested, no uncertainty exists as to the effectiveness of the inhibition on crack initiation. Examples are given where the CER test predicted immunity but the load relaxation test showed continued crack propagation.

When CER testing is used to predict or explain service failures, it is often criticized as being too severe. However, the paucity of information on SCC initiation mechanisms at smooth surfaces renders this a dangerous attitude. Many alloys carry films of reaction products developed over years of service, which are not easy to reproduce in the laboratory; these may facilitate SCC initiation in various ways during static loading [4]. In a recent study of the low temperature SCC of sensitized stainless steels, the effects of low levels of chloride ions were found to depend critically on the surface condition, even in CER tests [5]. Here we have compared results from static tests with the CER data, and have attempted to account for the observed differences in crack initiation behavior. In particular, the different effects of thiosulfate and tetrathionate on crack initiation are highlighted: this may be considered an important aspect of test methodology when SCC is known to have occurred as a result of an unknown combination of metastable sulfur compounds.

EXPERIMENTAL PROCEDURE

Material

Alloy 600 tubing, outside diameter 22.2 mm and wall thickness 1.24 mm, was obtained from Huntington Alloys (heat 9751). It had received a mill anneal and had the composition given in Table 1. The yield strength and

elongation to failure were about 310 MPa and 40%, respectively. 76 mm lengths of tube were cut and machined so that two curved tensile specimens could be obtained from each length by slitting lengthwise. The gauge length and width were 12.7 mm and 2.54 mm respectively. In addition, hemicylindrical tube sections were cut for use as U-bend specimens. For CER testing the tensile specimens were either sensitized directly at 621°C for 18 h or solution annealed at 1135°C for 45 min. before sensitization at 621°C for 18 h. The latter treatment gave a yield strength of about 210 MPa and an elongation to failure of about 54%. The sensitization heat treatment approximates to the stress relief treatment of the steam generators where the service failures occurred; when applied directly to the mill annealed material it did not affect the mechanical properties. U-bend tests were mainly carried out on directly sensitized material, with a few tests on samples which had been solution annealed prior to sensitization. The concave face of the tube was on the outside of the bend in every case, giving very severe stresses and a gradation in stress state.

Testing Environments

For CER testing in dilute thiosulfate solutions, the test environments were based on a 1.3% (0.21M) boric acid solution prepared from deionized water (conductivity < 0.1 μ S) and an analyzed grade of boric acid. The latter contained < 3 ppm Cl (giving < 39 ppb Cl⁻ in a 1.3% solution), < 1.6 ppm P as PO₄³⁻ (giving < 21 ppb P) and < 10 ppm S as SO₄²⁻ (giving < 130 ppb S or 4×10^{-6} M SO₄²⁻). Naturally these elements could be present in an insoluble form, giving even lower dissolved concentrations. Typically the 1.3% solution had a conductivity of 9.5 μ S at 25°C, with a pH close to 4.8. Lithium hydroxide, sodium thiosulfate and sodium tetrathionate were the other reagents used.

Constant Extension Rate Testing

The CER specimens, abraded to a 600 grit finish, were strained at a nominal rate of $3 \times 10^{-6} \text{ s}^{-1}$ using zirconia coated steel pins to transmit the load. About 0.3 mm of anomalous extension occurred in every test owing to lack of rigidity. In tests where the potential was monitored or controlled, an Inconel 600 wire was spot-welded to one end of the specimen as an electrical connection. A saturated calomel electrode (SCE) was placed inside a quartz tube with a capillary at one end plugged with glass wool, then immersed directly in the solution. This double diffusion barrier effectively prevented chloride contamination of the test solution. The solution was air-saturated and contained in a 3 liter beaker around the specimen assembly, with a Teflon lid. Temperature control was accomplished using a thermostatically controlled infra-red lamp. For tests where additions were made to the environment after crack initiation, a magnetic stirrer was used to ensure rapid homogenization. Stirring at low rates did not significantly affect crack growth. For interrupted tests, the machine was switched off and the decay of the load due to SCC monitored on a chart recorder. Changes in crack velocity due to environmental alterations were evidenced by changes in the rate of load decay. In one test, a crack was introduced at constant deflection by straining in a 0.1 M thiosulfate solution, then locking the machine. The test container was then flushed with progressively more dilute solutions until it contained 0.7 ppm sulfur as $\text{Na}_2\text{S}_2\text{O}_3$. The load decay was examined for evidence of continuing crack propagation; the effects of further additions of boric acid and lithium hydroxide were also investigated.

U-bend Testing

A wide variety of environments was used to test both solution annealed/sensitized (SAS) and mill annealed/sensitized (MAS) material. Most of the results will be reported in another paper [3]. Here only one set of tests is described: the evaluation of the relative effects of thiosulfate and tetrathionate on SCC susceptibility of MAS and SAS material.

RESULTS AND DISCUSSION

Microstructure of Material

It is worthwhile to review some of the factors influencing the sensitization of Inconel 600. Figure 1a shows the microstructure of the MAS material. This etch preferentially attacks carbides. Heavy grain boundary precipitation is accompanied by two types of intragranular carbides. Some are distributed rather randomly, while others are lined up along "ghost" grain boundaries. The latter are remnants of the structure prior to the mill anneal - indeed, the differences between the mill annealed structure (immune to SCC) and the MAS structure were very minor when examined at this resolution, so that the phosphoric acid etch is not a reliable indicator of sensitization in this material. The consequence of the limited carbon dissolution during the mill anneal is that the amount of carbon available for sensitization may be rather small (say, 0.01%) even in a 0.04% carbon material. Taken in conjunction with the variability in carbon content and mill anneal conditions of production tubing, these considerations rendered it necessary to use a solution anneal prior to sensitization in order to examine the environmental variables in SCC. The microstructure of the SAS material is shown in Figure 1b, and represents classical severe sensitization where almost the full 0.04% C was available in solid solution. In another paper (3) the results of electrochemical potentiokinetic reactivation (EPR) testing will be reported; when applied to MAS and SAS material this technique gave a good indication of the relative degree of sensitization. The charge densities per unit grain boundary area passed during reactivation of mill annealed, MAS and SAS material were 111, 209 and 774 C cm⁻² respectively. The electrolyte was 0.01 M KCNS + 0.5 M H₂SO₄ at room temperature, the passivation treatment 400 mV (SCE) for 4 minutes and the reverse scan rate 1 mV/s.

CER Testing

Preliminary tests on the MAS material using non-borated sodium thiosulfate solutions at 40°C showed that no SCC could be induced below 70 ppm S ($1.1 \times 10^{-3} \text{M Na}_2\text{S}_2\text{O}_3$). In a 0.1M solution cracking was rapid, with 0.7 mm crack growth in 24 h. Since these tests clearly offered no possibility of accounting for the steam generator failures, which occurred at much more dilute levels of sulfur, they were continued using SA3 material. Here, with 1.3% H_3BO_3 , SCC occurred at concentrations as low as 75 ppb sulfur ($1.2 \times 10^{-6} \text{M Na}_2\text{S}_2\text{O}_3$) and at high rates. The rate increased with temperature up to at least 80°C. All these results are discussed in detail elsewhere [2]. At 0.7 ppm sulfur the effects of thiosulfate and tetrathionate were comparable.

The influence of test procedure was revealed most clearly in tests where the effect of lithium hydroxide additions was studied. Although sufficient LiOH additions always inhibited SCC at a fixed sulfur level, an apparent increase in SCC susceptibility with Li concentration was observed in the range just below the critical inhibitory value. This effect is shown in Figure 2, for two SCC susceptibility indices, and was at first puzzling as the SCC process was thought to be influenced strongly by anion electromigration into the cracks. Additions of LiOH cause a proportional degree of ionization of the boric acid, giving HB_4O_7^- ions which carry current in competition with the thiosulfate ions and therefore restrict the thiosulfate enrichment which can be attained in a crack. One therefore anticipates that the ratio of inhibiting (HB_4O_7^-) to aggressive ($\text{S}_2\text{O}_3^{2-}$) ion concentrations will be more important than the absolute HB_4O_7^- concentration (i.e. the pH) in determining how much LiOH must be added to prevent SCC. A continuous decrease in SCC velocity with LiOH concentration is expected, other things being equal.

The effects of lithium hydroxide additions on propagating cracks were examined by interrupting a CER test and following the load decay with periodic LiOH additions (Figure 3). Here 5.6 ppm lithium again sufficed to lower the crack growth rate to an extremely low value at 0.7 ppm sulfur. Now, however, the cracking rate decreased smoothly with lithium concentration. For 7 ppm

sulfur approximately 150 ppm Li were required to inhibit SCC, and again the progression in rate was smooth. Again the most dramatic change in crack velocity occurred between Li/S ratios of 3 and 8. At low Li concentrations the cracking rate with 0.7 ppm sulfur was, surprisingly, higher than in CER tests (Figure 2). At the end of this test, examination of the specimen showed one deep, planar crack with several minor secondary cracks which could not have affected the load relaxation significantly. These results confirm the general expectations of the model based on electrotransport of aggressive and inhibiting anions.

It is notable how rapidly the environmental changes affected the cracking rate in the test shown in Figure 3. The notion that a stress-corrosion crack tip is isolated from the bulk environment is clearly wrong in this case. By including electrical forces in the analysis of mass transport in the cracks, the rapidity of the effect can be explained. For sensitized Type 304 stainless steel in non-borated thiosulfate solutions, the arresting of SCC by sulfate additions [6] is even more rapid owing to the higher crack velocity and consequently large potential gradient driving the sulfate ions into the crack and permitting a net diffusion of thiosulfate away from the crack tip.

The key to understanding the apparently conflicting results of Figure 2 lies in the influence of crack initiation frequency on some uncontrolled parameters in the CER test. As Figure 2 shows, additions of lithium hydroxide up to 2.8 ppm Li drastically reduce the number of cracks initiated during SCC. One effect of the reduced number of cracks could be to raise the crack tip strain rate compared with a multiple cracked specimen, thereby enhancing SCC. However, our interrupted CER tests in this system have shown rather little influence of stress on the crack growth rate, suggesting that the cracks are easily brought to their maximum velocity. This indicates a low apparent threshold stress intensity for SCC and a correspondingly low stress intensity at which the stage II (constant crack velocity) regime is reached. We therefore favor an electrochemical interpretation in which SCC propagation by anodic dissolution in the aerated solution generates a polarizing current which is proportional to the summed products of crack tip length and crack velocity. This current flows to the external specimen surface and depresses

the average potential of the specimen in an open circuit test. Depending on the impedance of the surface and the solution conductivity, the current flow may be more or less localized to the region around the crack. Since the crack velocity in dilute environments of this type increases with more positive potential over a large range [7], a specimen with many cracks will crack at a slower rate than one with one crack, other things being equal.

A typical potential versus strain trace for a CER test is shown in Figure 4. Initiation of SCC is characterized by a gradual shift of potential in the negative direction, which eventually stabilizes when a constant polarizing current is being generated by a fixed number of cracks moving at a constant rate. For this particular test, this regime occupies nearly half the total test duration. When final fracture occurs, the polarizing current from the cracks ceases and the potential rapidly returns to its value before crack initiation. In tests with many cracks, potentials as low as -300 mV (SCE) are obtained in lithium-free solutions and the cracking rate is restricted. In tests with only one crack of significant length, such as the interrupted CER test described earlier, the potential remains close to -100 mV and the cracking is much more rapid whether or not continuous straining is applied. Indeed, since further straining tends to initiate new cracks, the subsequent rate of cracking is more rapid in the interrupted (constant deflection) test than in the continuing CER test. The effect is particularly apparent in this material, as the currents emerging from cracks in sensitized alloys tend to be larger than in more homogeneous materials [6,8].

One might consider that the above effects could easily be eliminated by controlling the potential, and are therefore trivial. However, the solutions involved in these tests have very low conductivity and similar effects may operate in even more poorly conducting systems. Attempts to control the potential in very dilute solutions may create additional problems.

In Figure 5, the results shown in Figures 2 and 3 are constructed schematically, showing why the different test methods give different results.

The maximum crack velocities are drawn as varying linearly with potential, defining an ohmic resistance which is associated with the solution inside a crack; in practice, this varies in a very complex fashion because a faster moving crack attracts a more conducting solution. These considerations are discussed elsewhere in more detail [2]. For a fixed number of identical cracks there is a shift in potential depending on crack velocity, independent of the larger shift associated with differences in crack initiation frequency.

Correlation of Static and Dynamic Tests

In the preceding section it has been demonstrated that an initiated crack in SAS material can propagate with ease in environments representing the contaminated reactor water. However, even in CER tests initiation effects were important. In order to attempt a full description of the service phenomenon, it was necessary to study the initiation of SCC using U-bend specimens. All these tests were performed without removing the visible oxide film from the sensitizing heat treatment in air; thus the act of making the U-bend produces cracks in the film which should favor crack initiation. Similar flaws were probably present in the more complex surface film on the Inconel steam generator tubes, owing to high peak stresses during cooldown from high temperature.

In neutral solutions containing 0.1M $\text{Na}_2\text{S}_2\text{O}_3$ and at temperatures of 22, 40, 60 and 80°C, no SCC occurred in MAS U-bends for a test time of 2 weeks. This was not an especially surprising result, although the contrast with the 0.7 mm/day cracking rate in CER tests at 22°C is notable. In order to initiate SCC in 0.1M thiosulfate solutions at 22°C, it was necessary to bring the pH to 3-3.5 with sulfuric acid. Initiation occurred within a few hours, and the crack propagation rate was comparable with those measured in the CER tests. In another paper [6] it has been argued that the crack tip pH during thiosulfate SCC is close to 3; thus, in the acidified solution, the earliest stages of cracking are not hindered by the necessity for localized pH

changes. Parallel experiments with polished surfaces showed that the acidified thiosulfate solution caused a mild generalized intergranular attack in addition to the rapid SCC.

As supplied by most manufacturers, commercially pure sodium tetrathionate gives a pH of 3.5 - 4.0 for a 0.1M solution. This promotes very rapid SCC initiation and renders it an excellent test reagent for sensitization in both stainless steels and nickel alloys. This has been pointed out by Berge and Donati [9]. For the MAS U-bends, cracks were already visible after 1 hour immersion in aerated 0.1M $\text{Na}_2\text{S}_4\text{O}_6$ at 22°C. Further experiments with more dilute solutions showed that the tetrathionate could induce SCC of MAS U-bends in 4 days at a concentration (10^{-4}M) where thiosulfate was inactive even in CER tests. This is neither a pH nor a potential effect, but shows there are subtle differences between the metastable sulfur compounds in their ability to promote SCC initiation at smooth surfaces. In CER tests of sensitized stainless steels [10] or SAS Inconel 600 [2] thiosulfate causes SCC at lower concentrations than tetrathionate. For the 0.1M tetrathionate solution, deoxygenation to < 0.1 ppm by a nitrogen purge caused a drop in potential and a reduction in crack propagation rate by a factor of at least ten but did not eliminate SCC altogether. However, the result shows that the oxidizing power of tetrathionate is not the only factor determining the potential and crack propagation rate, as implied by Cowan and Gordon [11].

U-bend tests of SAS material produced cracking at aqueous sulfur concentrations close to those present in the reactor environment, but did not permit a proper evaluation of the various environmental factors involved. This emphasizes the unique value of the CER test. No cracking occurred for the water simulation of 1.3% H_3BO_3 , 0.7 ppm S (as $\text{Na}_2\text{S}_2\text{O}_3$) and 0.7 ppm Li (as LiOH) in a 4 week U-bend test, whereas in an interrupted CER test a 90% through-wall crack could be produced in 16 hours. For 1.3% H_3BO_3 with no lithium hydroxide additions, cracking occurred at ≥ 1.2 ppm sulfur whether added as $\text{Na}_2\text{S}_4\text{O}_6$ (10^{-5}M) or $\text{Na}_2\text{S}_2\text{O}_3$ ($2 \times 10^{-5}\text{M}$). Thus the large effect of the chemical state of sulfur is more noticable in the "less sensitized" MAS material than in SAS material.

Even in the MAS material most of the environmental effects observed in U-bend tests were exclusively associated with crack initiation. This was demonstrated by a composite experiment: a crack initiated by slow straining in 0.1M sodium thiosulfate at 22°C continued to propagate at constant deflection for 2 days in 1.3% H_3BO_3 + 0.7 ppm S (as $\text{Na}_2\text{S}_2\text{O}_3$). The cell was flushed repeatedly with the dilute solution. The average crack velocity following dilution was 1 nm s^{-1} , and lithium hydroxide additions inhibited SCC as before, showing that the crack tip environment had altered to that characteristic of 0.7 ppm bulk sulfur concentration. Thus one does not need to invoke metallurgical variability to account for the failure of the MAS tests to reproduce the steam generator cracking, provided that crack initiation was promoted in practice by some combination of factors not reproduced in the laboratory tests. Such factors include the special nature of the sulfur-rich surface film, which could locally generate aggressive metastable sulfur species such as polythionate ions.

Conclusions

1. Open-circuit CER testing in aqueous solutions may rank environments incorrectly owing to the influence of varying crack initiation statistics on the electrochemical potential of the specimen.
2. For sensitized Alloy 600 in dilute aerated thiosulfate solutions, the variation of open-circuit potential during a CER test is interpretable as an effect of currents emerging from growing cracks.
3. In this system, measurement of load relaxation following interruption of a CER test is a very rapid way of investigating inhibiting additions to the solution. These additions often alter the rate of crack propagation remarkably rapidly by influencing anion migration into the cracks.

4. Thiosulfate and tetrathionate ions, which cause similar rates of cracking in CER tests, differ greatly in their ability to promote smooth surface SCC initiation. These effects vary with the metallurgical condition of the alloy.

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Table 1 Analysis of Inconel 600 tubing

	Cr	Fe	C	Cu	Si	Al	Ti	Mn	Co	S	Ni
wt. %	14.62	9.41	0.04	0.40	0.32	0.29	0.22	0.17	0.02	0.001	balance

Figure captions

Figure 1 Microstructure of Inconel 600 tube material (electrolytic phosphoric acid etch)
(a) as received + 621°C for 18h
(b) 1135°C for 45 min. + 621°C for 18h

Figure 2 Effect of lithium hydroxide additions on SCC of solution annealed/sensitized Inconel 600 in air saturated 1.3% H_3BO_3 + 0.7 ppm sulfur as $\text{Na}_2\text{S}_2\text{O}_3$, 40°C. Average crack velocity and elongation to failure are shown for CER tests at $3 \times 10^{-6} \text{ s}^{-1}$ strain rate. For the interrupted CER test the crack velocity was estimated visually and from the linear load decay at constant deflection. Ductility loss is defined as (% elongation in air) - (% elongation in environment).

Figure 3 Load variation following interruption of CER test of solution annealed/sensitized Inconel 600 at 40°C. Initial solution was air saturated and contained 1.3% H_3BO_3 + 0.7 ppm sulfur as $\text{Na}_2\text{S}_2\text{O}_3$. Additions were made at times A to H, giving the compositions shown. The specimen contained one major crack.

Figure 4 Load and potential variation during a CER test of Inconel 600, solution annealed and sensitized, in air-saturated 1.3% H_3BO_3 + 7 ppm sulfur as $\text{Na}_2\text{S}_2\text{O}_3$ + 12 ppm Li as LiOH , 40°C. Strain rate = $3 \times 10^{-6} \text{s}^{-1}$. The extended elastic region is an artifact.

- A: establishment of passive state
- B: pre-initiation stage
- C: several cracks initiated
- D: several cracks in stage II growth giving constant polarizing current
- E: polarizing current due to crack tip dissolution ceases at specimen failure.

Figure 5 Schematic illustration of the variation of steady-state specimen potential (X, θ) with solution composition, crack velocity and number of cracks. Note that a CER test with 0.7 ppm S and no Li (many cracks) may give a lower mean cracking rate than a test with 2.1 ppm Li added (one crack).

Figure 1

(a)

(b)

Figure 2

FIGURE 3

FIGURE 4

FIGURE 5